CLIN 0014 Data Item

"Thermal In-Pouch Microwave Sterilization" Contract No. W911QY-09-C-0205

Data Item A0001 Contract No. W911QY-09-C-0205

Progress Reports

Printpack, Inc. Atlanta, GA

20120305091

DEFENSE TECHNICAL INFORMATION CENTER

Information for the Defense Community

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Quarterly Report

For the Period Ending
31 December, 2009
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Quarterly Report

For the period ending
31 December, 2009
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Summary: After one quarter, the project is on time and on budget with progress to report for each deliverable task. The first deliverable is ready to submit on schedule.

Project Overview

Objective

To advance the state of the art for Thermal Microwave Sterilization (MWS) for pouches of individual combat rations while maintaining current shelf life:

- improvements in the MWS process at Washington State University (WSU) and
- improvements in the non-foil light barrier materials considered in the Fy08 Printpack research

Deliverables

Task	Deliverable	Due date
1	10 Laminations	31 Dec 2009
2	Physical, Barrier, & Optical Data	28 Feb 2010
3	Photodegradation Data	31 Jan 2010
4	Retort & MWS Entrée Packages	30 Apr 2010
5	Hot Fill Packages	30 Apr 2010
6	Optimized MWS Entrée Packages	30 Jun 2010
7	MWS Validation Report	30 Jun 2010
8	Standard Condition Shelflife Modeling	30 Apr 2010
9	Extreme Condition Shelflife Modeling	31 May 2010
10	TTI Label Evaluation	30 Apr 2010
11	Printed Pouch TTI Evaluation	31 Aug 2010

(ToC)

Accomplishments

1. Task 1 (Laminations): As of the end of December, all laminations are completed; advances include new barrier materials and alternate opacifying pigments for sealant films. Details are provided in "Good News" section
2. Task 2 (Material data sheets): Additional Oxygen transmission rate measurement has been procured and installed in the Printpack Analytical Laboratory. Details are provided in "Equipment" section. The thin film dielectric property measurement technique developed at WSU during last's year's project continues to provide useful guidance about the materials' energy interactions in MWS. Values for each lamination made in Task1 will be provided with this method.
3. Task 3 (Photodegradation Data): Substantial resources have been dedicated to transferring the Virginia Tech photodegradation assessment technique to Printpack's Analytical Laboratory, including recruitment of a Ph.D. research specialist with solid phase micro extraction (spme) experience and procurement and installation of an autosampler for one of the lab's gas chromatograph/ mass spectrophotometers (GC/MS). Validation of the technique and calibration to the previous year's Virginia Tech results are underway. (Details are provided in "Good News" and "Equipment" section respectively.) Dr. Sean O'Keefe of the Virginia Tech Food Science Department, author of the previous year's report, has been very helpful and supportive of this effort.
4. Tasks 4,6,7 (MWS process): A scope of work as a subcontractor for this project has been approved with Professor Juming Tang of Washington State University (WSU). Details are provided in "Subcontracts" section Final administrative details for the subcontract are being finalized. We have agreed, with CFD coordination (Dr. Tom Yang), to use Chicken and Dumplings as the MRE ration to use for the validation report. WSU staff is already working to identify an appropriate inoculation technique for this complex protein, starch, vegetable entrée item.
5. Tasks 8,9 (Shelf life Modeling): A license has been signed for use of the "M-Rule Container Performance Model for Foods." (Initial training was conducted for the Printpack project team.)
6. Tasks 10,11 (TTI Technology): An initial specification for the performance envelop of Time Temperature Indicator has been negotiated with Segan Industries (Burlingame, CA). Subcontract details are currently in discussion. (Details are provided in "Good News" and "Subcontracts" section respectively.)

(ToC)

Risks

Technical and program Risks

Technical Risks

1. Task 1 (Laminations): (None…this task is effectively complete)
2. Task 2 (Physical, Barrier, Optical data):
    a. Two novel barrier films are added to the set used in the laminations from last year's project. Both provide improved water vapor barrier transmission rate (WVTR), but while compliant with US FDA requirements for high temperature cook-in processes, they are not intended for use at 121°C for the duration of a full retort cycle. *Risk mitigation:* To assess the thermal stability of laminations with these films, we will add WVTR and oxygen transmission rate (OTR) following thermal abuse to the flat and mechanically-abused barrier data provided for the other laminations. Filled Pouches will be processed at 121°C in the Clemson University Packaging Science Department's retort chamber for a full 40 minute retort cycle and for a 7 minute cycle to emulate the MWS process. Material from the treated pouches will be statistically analyzed to determine if WVTR and OTR are significantly affected. (Printpack's membership in the Clemson Center for Excellence in Flexible packaging- CEFPACK- will underwrite these studies).

    b. Alternate light barrier mechanisms are included in the present set of trial laminations. Last year's laminations utilized the intrinsic UV barrier of the films in the laminate plus visible light-opaque pigmentation in the adhesive layers to achieve maximum light absorption in the 200-700 nm range. The thin film MW resonance assessment technique developed by WSU in last year's project was able to measure MW absorbance differences among the previous set of trial laminations. In this year's trial laminations, pigmented sealant film layers are used. Some of these use US FDA compliant carbon black for pigmentation, others compliant "subtractive color system" colorants to block light. *Risk mitigation:* The battery of test methods demonstrated in last year's work, UV-vis spectrophotometry, surrogate photodegradation analysis, and MW resonance comparison will allow objective determination of the relative fitness for use of alternate light barrier materials with respect to shelf life, and MWS process efficiency.
3. Task 3 (Photodegradation Data)

    The interlaboratory transfer of Dr. O'Keefe's photodegradation technique to Printpack's lab could introduce an element of variability invalidating comparison of the two years' data. (O'Keefe's lab, rather than Printpack's did the previous work because of equipment gaps, and personnel skill neeedss). *Risk mitigation:* The equipment gap was closed with the installation of the spme autosampler at Printpack; the skills one, with the addition of a spme-experienced Ph. D.chemist to the lab staff. Even with this we recognized the need to validate the method practiced at Printpack with O'Keefe's. This has taken the form of a Printpack study to establish a

precision and accuracy level for the technique, to reduce the signal to noise ratio, and to confirm a limit of detection.

4. Tasks 4,6,7 (MWS process)

   Review of the previous US FDA validation of the WSU MWS process for mashed potatoes revealed the need to confirm that the packaging material used to contain the product did not adulterate it by leaching migrating chemicals in to food. *Risk mitigation:* Printpack will provide single-sided migration studies of the pouch materials used in last year's and this year's MWS chicken and dumplings. This experimental protocol will most likely conform to the US FDA "Chemistry Guidelines" for its "Food Contact Material Notification" program, but is subject to discussion with and concurrence by US FDA process authorities.

   The all-plastic laminated material used to make the pouches for last year's Chicken and Dumpling entrée presented a spotted pattern of small voids visible from the outside of the pouches. While not affecting the functionality of the material, the effect was definitely an unacceptable defect that is not acceptable in the future. *Risk mitigation:* Printpack has initiated a statistical analysis of laminator operating conditions intended to isolate the critical variables responsible for the defect. Additionally, mechanical modifications have been designed to widen this window of operability. These will be installed in January, 2010 at which time another statistical analysis will confirm process capability.

   The WSU MWS process for mashed potatoes defined an acceptable method for inoculating the product with appropriately thermal resistant-spores subject only to placement at the cold spot of the tray/lidding package. While identifying the cold spot in a pouch is a similar process, each of the elements of the diverse chicken and dumpling food product has its distinct thermal heating properties. MWS process validation requires: convincing assessment of the combination of food component/pouch position that is least susceptible to heat sterilization; spore inoculation of that position, and incubation of pouches to determine if those spores survived a given thermal treatment. *Risk mitigation:* WSU personnel have already analyzed the pouches used to contain last year's MRE entrees to identify the cold spot in a homogenous product. They have also assembled thermal data on the product's components and have now determined that the dumplings represent the least thermally susceptible component, as a consequence of both their size and thermal properties. With this early identification of critical process validation elements, they will be able to develop inoculation techniques that will satisfy US FDA requirements.

5. Tasks 8,9 (Shelf life Modeling)

   The M-Rule Container Performance Model for Foods has been developed and validated foods and packaging materials on less complex than those involved in shelf stable combat rations. Its adoption to these systems cannot be claimed to forecast shelf life performance until experimental data is

developed for comparison to predicted results. *Risk mitigation:* Printpack will:

- Establish the database for its packaging materials in the M-Rule Model format.
- Define "idealized" foods, representing MRE entrees, hot fill sauces, and dessert bars, in the context of the model's key variables (water activity, vitamin content, oxidative vulnerabilities, etc.)
- Model primary oxidative and moisture dynamics within packaging materials under standard and extreme storage scenarios to provide a sensitivity analysis of packaging material and product variables most critical to shelf life limits.

6. Tasks 10,11 (TTI Technology)

   The time temperature indicator system used to print an indicator message on packaging material must resist not only premature development of indicia during package material conversion, material shipment and storage, product packaging and sealing but also early message fade during post-processing shipment and storage. *Risk mitigation:* Printpack and Segan have agreed to evaluate three Segan chemistries in the label (Task 10) phase. The WSU thermal cell in oil bath technique will be adapted to quantify and compare the color change kinetics of the three chemistries. This information will be used to select a chemistry to be adapted to a thermally resistant flexographic ink used to print laminations used to make pouches.

Program Risks

7. Task 1 (Laminations): (None…this task is effectively complete)
8. Task 2 (Physical, Barrier, Optical Data): As we found last year, OTR and WVTR measurement for the high barrier laminations is the rate limiting work step for this task. With all laminations complete at the end of December, we can estimate elapsed time required to obtain all measurements, and decide to outsource them if necessary.
9. Task 3 (Photodegradation Data): With the positive results to date in transferring the Virginia Tech methodology to the Printpack lab, we recognize little to no program risk for this task.
10. Tasks 4,5,6,7 (Packaged Products): Timely completion of this task is contingent on coordination of the availability of Printpack's packaging material with product (entrée, sauce, and dessert bar) processing. In the former area, we have advised suppliers of the expected optimum raw materials of our planned orders for delivery in late spring and are confident of timely production and availability of the laminations. For the latter area, we would expect to utilize the same military contractors (Thermo Pac and Sterling Foods) used in the previous year's program. Entree production is contingent on WSU resources. We expect regular, at least weekly communication with WSU staff regarding plans and scheduling of entrée

preparation, packaging, and processing. WSU administration has committed to maintain the WSU MWS process pilot plant at its present location through the end of December, 2010. Additionally, Printpack plans to participate in the commercially-oriented Microwave Consortium being organized to scale up the technology from this pilot scale.

11. Tasks 8,9 (Shelf life Modeling): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.
12. Tasks 10,11 (TTI Technology): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.

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Issues

Unexpected Issues

TASK AREA	COMMENT
1. Task 1 (Laminations):	(none)
2. Task 2 (Barrier data):	(none)
3. Task 3 (Photodegradation Data)	(none)
4. Tasks 4,6,7 (MWS process)	The need to provide data confirming that the MWS process itself does not cause chemical changes in packaging materials that are otherwise compliant with US FDA requirements for high temperature sterilization was not recognized in the original project scope. Directed studies to provide such data (according to US FDA protocols*) are readily available through industry laboratories and will be secured by Printpack for both FY2008 and FY2009 pouch stock.
5. Tasks 8,9 (Shelf life Modeling)	(none)
6. Tasks 10,11 (TTI Technology)	(none)

*
http://www.fda.gov/Food/GuidanceComplianceRegulatoryInformation/GuidanceDocuments/FoodIngredientsandPackaging/ucm081818.htm. See especially "APPENDIX II. SELECTED MIGRATION TESTING PROTOCOLS"

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Details

Good News

TASK AREA	COMMENT
1. Task 1 (Laminations):	• New improved WVTR films from Toppan (GL-ARHF) and SKC (SX03 Y07) have been used in the trial laminations • New light barrier techniques have been used in the trial laminations. One of these utilizes food contact grade carbon black; the other comprises a coextruded film using individual pigmented layers of cyan, magenta, and yellow (the "subtractive colors") and a final reflective layer of white • The effect of these innovations on WVTR and opacity respectively will be quantified and reported in the next quarterly report.
2. Task 2 (Barrier data): 3. Task 3 (Photodegradation Data)	• Both of these efforts have been expedited by the early installation of supporting analytical equipment at Printpack.
4. Tasks 4,6,7 (MWS process)	• The WSU MWS process has received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding. (Annex A)
5. Tasks 8,9 (Shelf life Modeling)	Licensing and training are complete
6. Tasks 10,11 (TTI Technology)	A working specification for the TTI technology has been prepared. (Annex B)

(ToC)

Details

Technical

TASK AREA	COMMENT
7. Task 1 (Laminations):	▪ Laminations complete and ready to submit. Detailed listing in Annex D
8. Task 2 (Barrier data): 9. Task 3 (Photodegradation Data)	▪ Both of these efforts have been expedited by the early installation of supporting analytical equipment at Printpack. ▪ Printpack's Analytical Lab is currently finishing validation of its photodegradation assessment methodology based on last year's Va Tech report. (Annex F) ▪ WSU has confirmed that its thin film MW resonance test provide valid material distinctions
10. Tasks 4,6,7 (MWS process)	▪ The WSU MWS process has received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding. (Annex A) ▪ Printpack ha completed a designed experiment with its commercial intended to identify the root cause(s) of the "pocked" appearance of laminations made last year. (Annex E)
11. Tasks 8,9 (Shelf life Modeling)	▪ Work will begin in January, 2010
12. Tasks 10,11 (TTI Technology)	▪ Confirmed the ability to test the performance of the TTI technologies (at the label stage) with the WSU Test Cell technique, allowing rapid screening of options and potentially development of a full kinetics description of the TTI color change.

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Details

Financial

Project Expenses as of 26 Dec 2009

COST ELEMENT	Contract Amt	Qtr 1 Amt
Total Direct Labor	232,930	28,496
Payroll Tax & Benefits	84,511	10,339
Departmental Overhead	135,996	16,637
Labor Total	453,437	55,472
Consulting & Services	544,500	0
Materials	70,850	33,170
Travel	24,530	6,660
Other Direct Costs	160,760	99,505
Subtotal Costs	1,254,077	194,807

Project expenses to date as captured in Printpack's financial accounting system indicate that the work remains on budget. The relatively low expense amount to date reflects the lack of any payments made to the two expected subcontractors and captured in the system. However, in both cases, subcontract work has begun, and the record of payments will reflect this work and more by the next quarterly report

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Equipment

Equipment	Cost	Assoc. Task(s)	Location
OXTRAN® 2/21 SL Env. Chamber Operating. System	$61,641	2	Printpack Analytical Services Lab 5 Barber Industrial Ct. Villa Rica, GA 30180
Leap Autosampler System	$36,107.	3	

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Subcontracts

Subcontractor	(Est)Cost	Assoc. Task(s)	Status
Washington State University	$400,550	4,6,7 See "Annex C"	Scope & price negotiated/ final draft in negotiation
Segan Industries	$175,000.	10.11 See "Annex B"	Scope negotiated/ draft in negotiation

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Annex A: Announcement of USFDA Validation of WSU MWS Process

FDA Approves WSU Researcher's Revolutionary New Food Processing Technology

Wednesday, Oct. 28, 2009

http://www.wsunews.wsu.edu/pages/publications.asp?Action=Detail&PublicationID=16596

PULLMAN, Wash. - Imagine a salmon filet that looks, tastes and is as nutritious as freshly cooked salmon but has a shelf-life of more than six months. A new technology developed at Washington State University will make that dream a reality.

For the first time ever, the U.S. Food and Drug Administration has approved the use of microwave energy for producing pre-packaged, low-acid foods, a major milestone that clears the way for its commercialization. The technology developed at WSU could revolutionize how we preserve and process food.

Juming Tang, a professor in the WSU Department of Biological Systems Engineering, led a team of university, industry and U.S. military scientists to develop the technology. The outcome results in food with a longer shelf life as well as better flavor and nutritional value compared to more traditional food processing methods such as canning.

"New processes for producing shelf-stable, low-acid foods must pass rigorous reviews by FDA to ensure that the technology is scientifically sound and the products will be safe," Tang said. "Our team patented system designs in October 2006 after more than 10 years of research. We spent another three years, developing a semi-continuous system, collecting engineering data and microbiologically validating the process before receiving FDA acceptance."

The team's Microwave Sterilization Process technology immerses the packaged food in pressurized hot water while simultaneously heating it with microwaves at a frequency of 915 MHz — a frequency which penetrates food more deeply than the 2450 MHz used in home microwave ovens. This combination eliminates food pathogens and spoilage microorganisms in just five to eight minutes and produces safe foods with much higher quality than conventionally processed ready-to-eat products.

Spearheaded by C. Patrick Dunne, Department of Defense combat feeding directorate at the U.S. Army Soldier Systems Center at Natick, Mass., the project has been funded from a variety of sources and a consortium of industry members that include Kraft Foods, Hormel, Ocean Beauty Seafoods, Rexam Containers, Ferrite Components and Graphic Packaging. The WSU team also worked closely with process authorities of the Seafood Products Association in Seattle and Hormel to establish validation procedures and in preparation of filing documents. In addition, faculty members from other WSU departments, particularly Food Science, contributed to the success of the project.

"The team's collective efforts have brought a new food processing technology to the forefront which will truly benefit not only the commercial sector but our war-fighters worldwide with a wider variety of high quality, shelf-stable foods," said Gerald Darsch, director of the U.S. Department of Defense Combat Feeding team. "It is truly a tremendous accomplishment."

Evan Turek, senior research fellow at Kraft Foods, said Tang's new technology will make a huge difference for the food industry.

"Since the introduction of industrial microwave ovens in the late 1940s, the food industry has been interested in exploiting the rapid heating capability of microwaves to

improve the quality of canned food," he said. "The technical issue has always been ensuring uniform and reproducible heat treatment. Dr. Tang had a vision about how this might be overcome, and with his leadership and the engineering prowess of his research staff and students, a protocol for practicing and validating microwave sterilization was established. Kraft Foods is proud to have been an early supporter of the research program at WSU and looks forward to the commercialization of the technology."

WSU officials agreed.

"This is a great example of how research universities like Washington State University produce breakthroughs that make an immediate impact on our nation and world. This new technology promises significant advances in food safety and quality to benefit everyone," said Howard Grimes, vice president for research.

Dan Bernardo, dean of the WSU College of Agricultural, Human, and Natural Resource Sciences, said the impact of the science will be dramatic.

"There have been very few advances leading to FDA accepted food processing technologies in recent history," he said. "The FDA's approval of this new technology truly could revolutionize the way we process and preserve food, ensuring food safety, increasing its longevity and maximizing the retention of its flavor and nutrition."

Ralph Cavalieri, director of the WSU Agricultural Research Center, said Tang's research has global benefits.

"It is important across a range of applications," he said, "from feeding astronauts on long-term space missions or soldiers in the field to transporting and storing food to areas of the world where people are unable to produce enough food locally to feed themselves."

Cavalieri said the project would not have been possible without support from a variety of sectors.

"We have worked synchronously with industry, the army and the university to make this happen," he said. "Dr. Tang's research also has received incredible support from Washington's Congressional delegation, especially Sen. Patty Murray."

Sen. Murray said ensuring funding for projects such as Tang's is part of an overall effort to support Washington's agricultural and food industry in ways that benefit the nation and world.

"This is great news for WSU, our growers and American food processors," she said. "It will help our growers and processors stay more competitive in the global marketplace and increase food safety for consumers

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Annex B: Working Specification for TTI

TTI Design Criteria: Microwave Thermal Sterilization Process

Print/Processing Conditions	Duration	Exposure	State Change	Pressure
Packaging Material				
Flexographic print/drying:	less than 1 second	80 – 100°C	reversible	Ambient
Nip press heat lamination:	less than 1 second	80 – 90°C	reversible	20-60 psi
Storage stability:	3-6 Months	20 – 25°C	reversible	Ambient
Product & Processing				
Hot filling	10 – 30 minutes	70 – 90 °C	reversible	Ambient
Pre warming	10 – 30 minutes	70 – 100°C	reversible	Ambient
Microwave sterilization:	3-5 Min.	120 – 130°C	irreversible	30 psig
Post processing storage	2 years	0 – 60°C	no reversion	Ambient

MTSP Sensing Composition Criteria: Working Criteria

- Compatible with flexographic printing on oriented Polyester film: solvent or aqueous based without use of "hazardous air pollutants" *http://www.epa.gov/ttn/atw/188polls.html*
- Survive reversible conditions up to 100°C and becomes irreversible above 120°C
- Must survive pressure, lamination step, and chemical interaction with laminate
- Visually addressable after complete cycling and readable after package cools
- Microwave compatible – responds to conductive heat from heated product not microwave energy, no metal allowed
- Must not revert during post storage conditions of 3 years at 27°C (80°F) or 6 months at 38°C (100°F) [i.e. until consumption]

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Annex C: WSU Subcontract Budget

Juming Teng application to Springpeck
July 1, 2010 - June 30, 2011
John Anderson
335-6642; fax 335-2722
jre@wsu.edu

PI's Name:						Year 1
Agency:						
SALARIES - 01	**Pay Rate**	**# Mos.**	**% FTE**			
Fang Liu	Personal & Confidential				**Salary**	29,151
				Benefits	32.00%	9,328
Galina Mikhaylenko					**Salary**	17,879
				Benefits	42.00%	7,509
Jae Hyung Mah					**Salary**	19,470
				Benefits	43.00%	8,372
Zhongwei Tang					**Salary**	20,520
				Benefits	41.00%	8,413
Juming Tang					**Salary**	16,508
				Benefits	29.40%	4,853
PhD Student			1		**Salary**	14,734
					QTR	8,105
					Health	1,613
					1.5%	221
Master Student			0		**Salary**	-
					QTR	-
					Health	-
					1.5%	-
WAGES - 01	**$ Per Hr.**	**Hrs/Wks**	**# Wks.**			
Student (full-time):	$0.00	0	0		**Wages**	-
				Benefits	2.1%	-
Student (part-time):	$20.46	20	13		**Wages**	5,320
(graduate student in summer)				*Benefits*	9.7%	516
					Total Salary	118,262
					Total Wages	5,320
					Total Salary & Wages	123,582
BENEFITS - 07						
					Total Benefits	48,930
					Total Salaries/Wages/Benefits	172,512
CAPITAL EQUIPMENT - 06 ($5,000 +)						
Residue gas measurement device						8,000
Network Analyzer						37,000
Single-mode resonance cavity for package material dielectric property measurement						7,000
					Total Capital Equipment	45,000
GOODS/SERVICES - 03						
Rental for FCI generator, directional couplers, waveguide						57,500
Materials and chemicals						4,000
Fiber optic sensors, Ellab sensors, replacement parts						3,000
Off-Site Rental						
Non-Capital Equipment (Under $4,999)						
Other						
					Total Goods/Services	64,500
TRAVEL - 04						
Domestic						3,500
Foreign						
					Total Travel	3,500
TOTAL DIRECT COSTS						285,512
EXCLUSIONS						
QTR						8,105
Equipment (Over 5k)						45,000
Subcontracts (After Initial $25K For Each Subcontract)						-
Other (Off-Site Rental & Stipends, Etc)						
					Base	232,407
			F&A Rate	**MTDC**	49.5%	115,041
			F&A Rate	**Total Cost**	0.0%	-
			F&A Rate	**Total Dir**	0.0%	-
TOTAL F&A - 13						115,041
TOTAL COSTS						400,553
F&A Base Type:	MTDC	TD	TC	SWB	Other	
	X					

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Annex D: 10 Barrier Laminations

Lam Number	Outer Layer	Barrier Layer	barrier2 Layer	Sealant Layer	COMMENTS
1*	OPET	KUR-C	KUR-N	Cblack B343T-997	Carbon Black Sealant w/ opaque adhesive
2*	OPET	KUR-C	KUR-N	CMYW B343T-997	CMYW Black Sealant w/ opaque adhesive
3*	OPET	KUR-C	KUR-N	Clear B343T-997	Clear Sealant w/ 2 opaque adhesives
4*	OPET	KUR-C	KUR-N	Cblack B343T-997	Carbon Black Sealant
5#	OPET	GL-PET-ARH	KUR-N	Clear B343T-997	Retort Grade GL OPET
6#	OPET	GL-PET-ARHF	KUR-N	Clear B343T-997	Sub-Retort Retort Grade GL OPET
7#	OPET	SX03 Y07	KUR-N	Clear B343T-997	SKC PvDC_Ctd Barrier OPET
8^	OPET	GL-PET-ARHF	BON	Clear B343T-997	GL Barrier only
9^	OPET	KUR-C	BON	Clear B343T-997	Kurarister-C Barrier 4-ply lamination
10^	OPET	KUR-C	n/a	Clear B343T-997	Kurarister-C Barrier 3-ply lamination

*: Alternate opaque sealant.
#: Control and better WVTR
^: Lower cost alternatives
Adhesive
Clear
Pigmented

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Annex E: DOE for Buckling Reduction

Buckling is defined as web wrinkling in the cross direction (CD) of a web. The causes of buckling are great and varied including defective raw material (baggy, wrinkled, etc), machine misalignment and air entrapment at the rewind. In this Design of Experiment (DOE) we are looking at factors that we have the most control of in our converting facility, namely air entrapment at the rewind (machine alignment will be addressed with installation of the outfeed nip). The four main inputs in rewinding were selected as variables for the experiment.. We performed two sets of experiments utilizing both fiber and steel cores to cover the wide range of structures and machine conditions that we run.

Minitab was used to both design the experiment and analyze the data. A 2-level factorial DOE was used with 4 variables, resulting in 9 sets of run conditions for each type of core. This balanced both practicality of the trial with sufficient resolution to obtain statistically significant results. Trials were run on consecutive days holding all machine conditions constant including staffing to minimize variability. Data was gathered watching each roll as it was unwound on the slitters. Buckling severity was gauged and recorded for each master roll, noting both extent and severity of buckles. A scale was created from 0 – 5 with 0 being no buckling observed to 5 being severe buckling from core to top of roll and extending across the face of the roll (See Appendix A).

The Main Effects plots (Appendix B) from Minitab show the individual contributions of each factor to buckling. These plots seem to mirror each other, except in the Variable D plot. Upon further research and analysis, I found two distinct factors that contributed to the differences: taping of cores and machine capability. First, the fiber cores were taped in a spiral direction that at certain conditions failed to adhere to the core and caused buckling from uneven transfer of the web. Also, the machine is not capable of running the full range of settings for variable D. Conclusions were made that the greatest factor influencing buckling is variable A, followed by Variable B, to some extent variable C and lastly, variable D.

The Interaction plots show the most effective combinations of each factor in creating or reducing buckling. These plots showed the dominance of Variable B over all other factors in regards to buckling. This is very evident in the steel core trials where the machine's limit for variable B was applied. The results are conclusive that maximizing variable A and variable B were most effective in reducing buckling.

One factor that was found to be significant during this trial, yet not relevant to machine conditions is the raw material. We witnessed varying degrees of buckling that could be directly correlated to the profile of the sealant film we were running. Baggy edges or lanes were prone to buckling, especially on low tension runs. We were however able to minimize the effects of the profile issues with certain process conditions..

In general, increasing variable A and variable B are the most effective means of reducing buckling. To a lesser extent, decreasing machine variable C can also have a positive impact on buckling, but as a last resort when maximum feasible levels for variable A and variable B are reached for any given structure. While these results do show us the means by which we can reduce buckling, they do not give us the exact settings for every structure to eliminate buckling. By utilizing our Standard Operating Conditions

(SOC's) and applying these findings we should be able to minimize buckling on the adhesive laminator and other similar pieces of equipment.

Appendix A – Parameters and Results

Fiber Core:

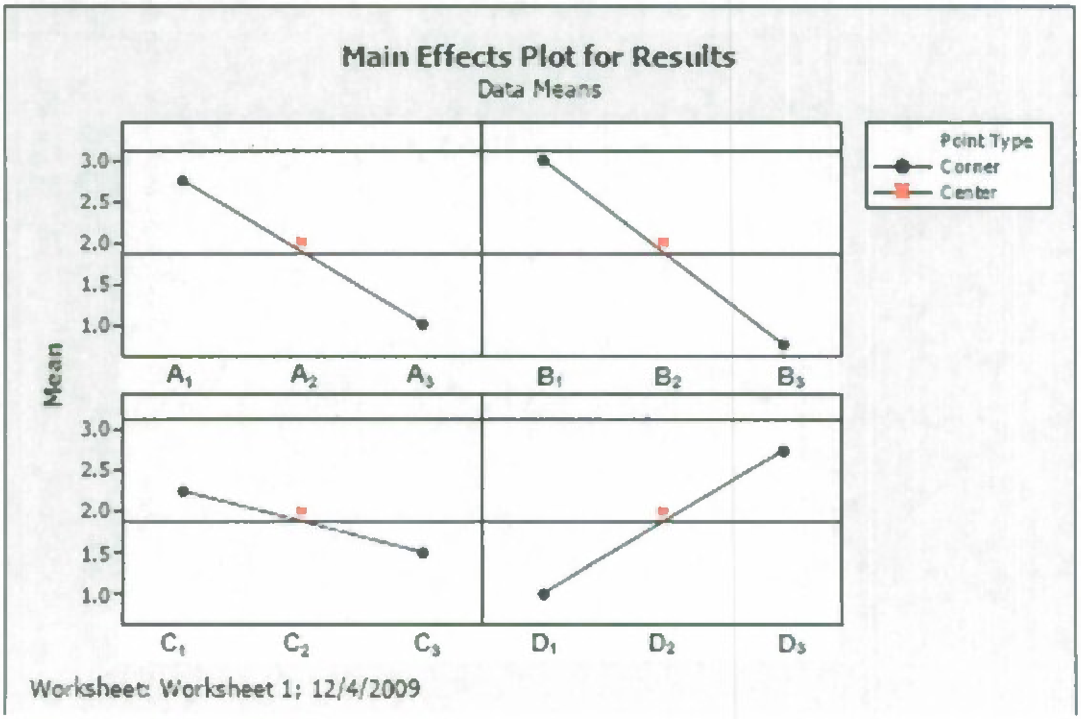
Run Order	Variable A	Variable B	Variable D	Variable C	Results
1	1	1	1	1	3
2	3	3	3	3	0
3	2	2	2	2	2
4	3	1	3	1	0
5	1	3	1	3	2
6	3	3	1	1	0
7	1	3	3	1	1
8	3	1	1	3	4
9	1	1	3	3	5

Fiber Core:

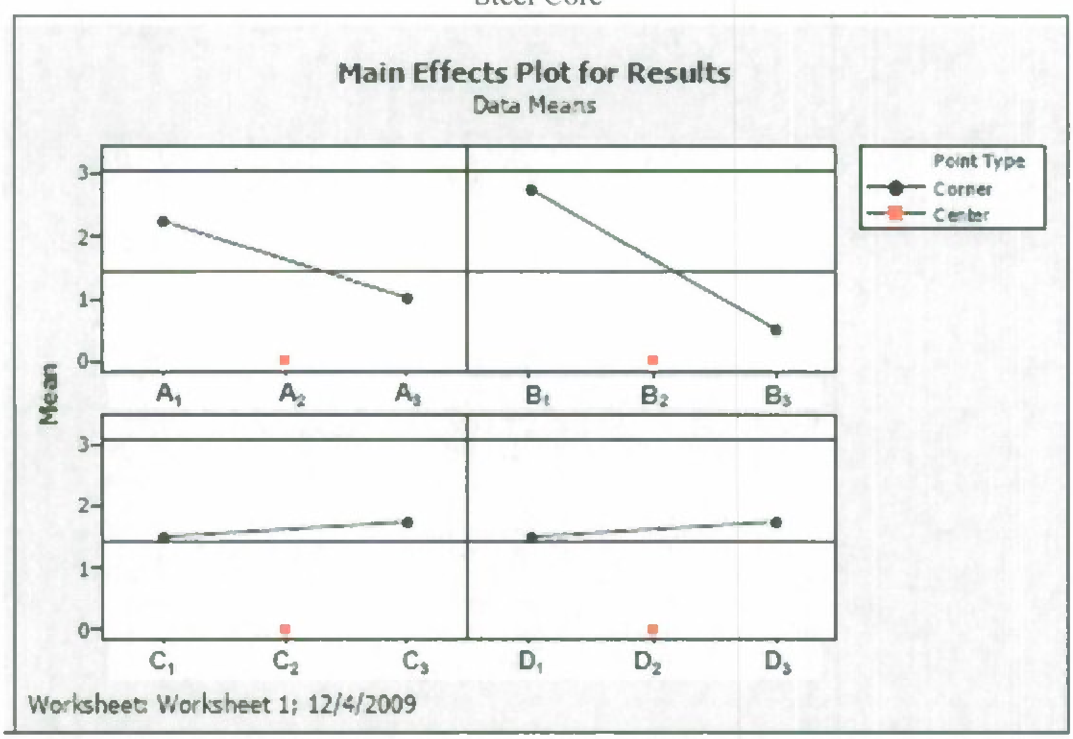
Run Order	Variable A	Variable B	Variable D	Variable C	Results
1	1	1	1	1	3
2	6	3	3	3	0
3	2	2	2	2	0
4	6	1	3	1	2
5	1	3	1	3	1
6	6	3	1	1	0
7	1	3	3	1	1
8	6	1	1	3	2
9	1	1	3	3	4

Appendix B – Main Effects Plots

Fiber Core

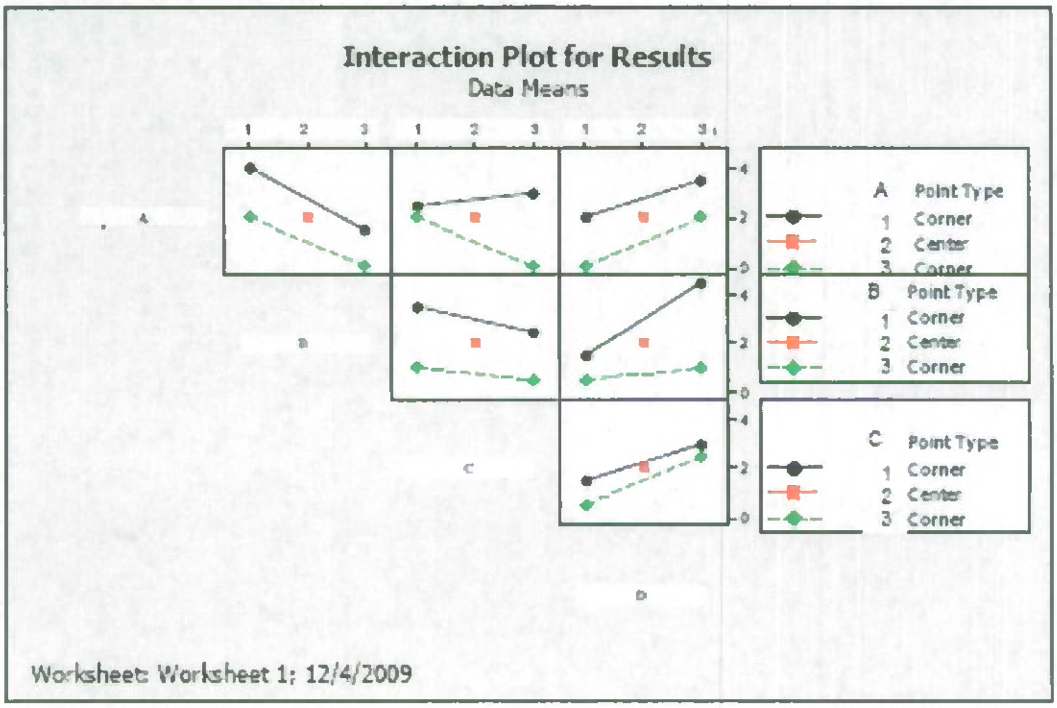


Steel Core

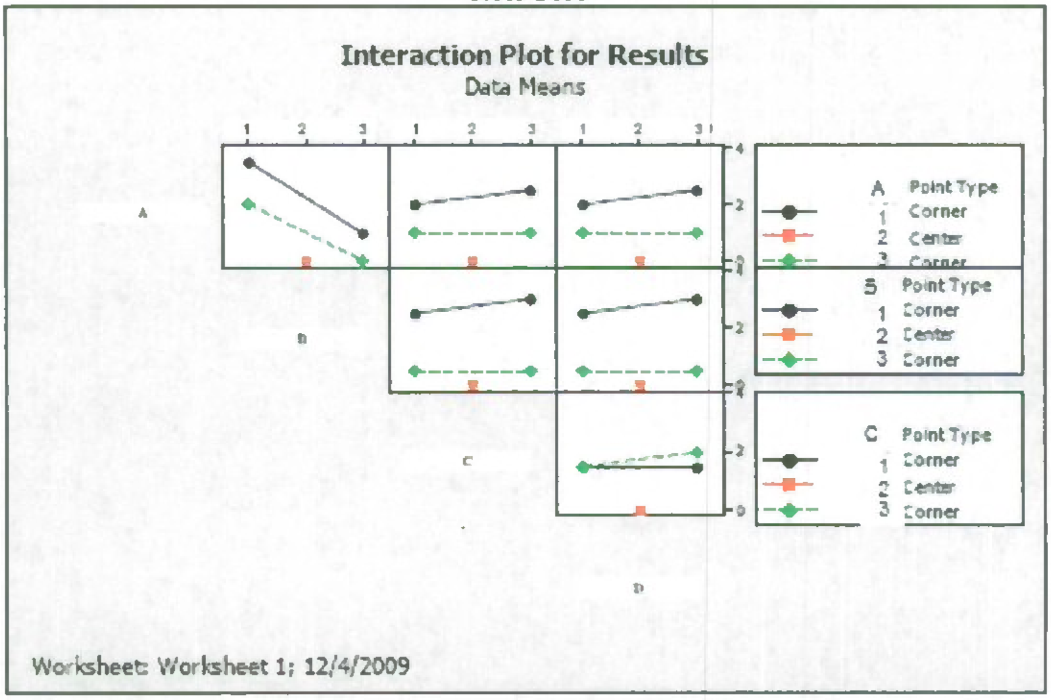


Appendix C – Interaction Plots

Fiber Core



Steel Core



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ANNEX F: SPME-GC-MS Method

Gas Chromatograph: HP-Agilent 6890N

- Column: Agilent Carbowax 20M, 30 x 0.25 x 0.25
- Carrier gas: Helium 1.5 ml / min, constant flow mode
- Inlet temp: 270 °C
- Injection mode: splitless
- Oven:
  1. 40°C (1 min hold)
  2. 5°C gradient / per min to 200°C (hold 2 min)
- Auxiliary line to MSD: 210°C

Detector: HP-Agilent 5973 Mass Selective Detector (MSD)

- Scan range *m/z* 50-550
- Autosampler: LEAP Technologies CombiPal

SPME fiber: DVB/CAR/PDMS

- Extraction time: 15 min with agitation
- Extraction temp: 60°C

Validation to include:

- linear dynamic range,
- precision,
- accuracy,
- recovery (function of the amount of yogurt added to vial),
- specificity

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Quarterly Report

For the Period Ending
31 March, 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Quarterly Report

For the period ending
31 March, 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Summary: After two quarter, the project is on budget with slippage in some early tasks. The deliverables 1 and 3 have been submitted. Significant progress for deliverables 2 and 4 through 7 is reported here.

Project Overview

Objective

To advance the state of the art for Thermal Microwave Sterilization (MWS) for pouches of individual combat rations while maintaining current shelf life:

- improvements in the MWS process at Washington State University (WSU) and
- improvements in the non-foil light barrier materials considered in the YPrint-pack research

Deliverables

Task	Deliverable	Plan date	Act/Ant date
1	10 Laminations	31 Dec 2009	08 Jan 2010
2	Physical, Barrier, & Optical Data	28 Feb 2010	30 Jun 2010
3	Photodegradation Data	31 Jan 2010	31 Mar 2010
4	Retort & MWS Entrée Packages	30 Apr 2010	30 Jun 2010
5	Hot Fill Packages	30 Apr 2010	30 Jun 2010
6	Optimized MWS Entrée Packages	30 Jun 2010	31 Jul 2010
7	MWS Validation Report	30 Jun 2010	31 Jul 2010
8	Standard Condition Shelflife Modeling	30 Apr 2010	31 May 2010
9	Extreme Condition Shelflife Modeling	31 May 2010	31 May 2010
10	TTI Label Evaluation	30 Apr 2010	31 May 2010
11	Printed Pouch TTI Evaluation	31 Aug 2010	31 Aug 2010

(ToC)

Accomplishments

1. Task 1 (Laminations): All laminations are completed; including advances include new barrier materials and alternate opacifying pigments for sealant films. (Details are summarized in ANNEX A".)
2. Task 2 (Material data sheets): Additional Oxygen transmission rate measurement has been procured and installed in the Printpack Analytical Laboratory. Details are provided in "Equipment" section. High barrier performance of materials results in lengthy cycle times for each measurement of OTR and WVTR. Only these data remain for completing this task (Available Data in ANNEX B-D.) The thin film dielectric property measurement technique developed at WSU during last's year's project continues to provide useful guidance about the materials' energy interactions in MWS. Values for each lamination made in Task1 have been developed with this method. (Data in ANNEX E)
3. Task 3 (Photodegradation Data): the Virginia Tech photodegradation assessment technique has been transferred to Printpack's Analytical Laboratory. Validation of the technique and calibration to the previous year's Virginia Tech results proved difficult because experimentally detected levels are at or below the limits of detection. Dr. Sean O'Keefe of the Virginia Tech Food Science Department, author of the previous year's report, has been very helpful and supportive of this effort. (Data in ANNEX E.)
4. Tasks 4, 6, 7 (MWS process): The subcontract with Professor Juming Tang of Washington State University (WSU) for this project has been signed. Details are provided in "Subcontracts" section. We have agreed, with CFD coordination (Dr. Tom Yang), to use both Chicken and Dumplings and a salmon filet wih Alfredo sauce as the MRE rations to use for the validation report. WSU staff has worked to identify relevant dielectric data, heating profiles, and inoculation methods.
5. Tasks 8 &9 (Shelf life Modeling): Validation of loaded data and will take longer than anticipated for the initial phase (standard distribution.) However, this validation allows the second phase (extreme distribution systems) to proceed very quickly following the initial one.
6. Tasks 10 & 11 (TTI Technology): A subcontract with Segan Industries (Burlingame, CA) for the development of a flexographic ink providing Time Temperature Indications on microwave sterilized pouches has been signed and investigations begun.

(ToC)

Technical and program Risks

Technical Risks

1. Task 1 (Laminations): (None…this task is effectively complcte)
2. Task 2 (Physical, Barrier, Optical data):
   a. Two novel barrier films are added to the set used in the laminations from last year's project. Both provide improved water vapor barrier transmission rate (WVTR), but while compliant with US FDA requirements for high temperature cook-in processes, they are not intended for use at 121°C for the duration of a full retort cycle. *Risk mitigation:* To assess the thermal stability of laminations with these films, we will add WVTR and oxygen transmission rate (OTR) *following thermal abuse* to the flat and mechanically-abused barrier data provided for the other laminations. Filled Pouches will be processed at 121°C in the Clemson University Packaging Science Department's retort chamber for a full 40 minute retort cycle and for a 7 minute cycle to emulate the MWS process. Matcrial from the treated pouches will be statistically analyzed to determine if WVTR and OTR are significantly affected. (Printpack's membership in the Clemson Center for Excellence in Flexible packaging- CEFPACK- will underwrite these studies). *This effort will be initiated when the current backlog of barrier testing is relieved.*
   b. Alternate light barrier mechanisms are included in the present set of trial laminations. Last year's laminations utilized the intrinsic UV barrier of the films in the laminate plus visible light-opaque pigmentation in the adhesive layers to achieve maximum light absorption in the 200-700 nm range. The thin film MW resonance assessment technique developed by WSU in last year's project was able to measure MW absorbance differences among the previous set of trial laminations. In this year's trial laminations, pigmented sealant film layers are used. Some of these use US FDA compliant carbon black for pigmentation, others use compliant "subtractive color system" pigments to block light. *Risk mitigation:* The battery of test methods demonstrated in last year's work, UV-vis spectrophotometry, surrogate photodegradation analysis, and MW resonance comparison will allow objective determination of the relative fitness for use of alternate light barrier materials with respect to shelf life, and MWS process efficiency. *Initial assessment of transmittance, photo-oxidation, and dielectric properties data suggests that "olive-drab" pigmented adhesive is adequate for the all-plastic light barrier structures.*
3. Task 3 (Photodegradation Data)

   The interlaboratory transfer of Dr. O'Keefe's photodegradation technique to Printpack's lab could introduce an element of variability invalidating comparison of the two years' data. (O'Keefe's lab, rather than Printpack's did the previous work because of equipment gaps, and personnel skill needs). *Risk mitigation:* The equipment gap was closed with the installa-

tion of the spme autosampler at Printpack; the skills one, with the addition of a spme-experienced Ph. D. chemist to the lab staff. Even with this we recognized the need to validate the method practiced at Printpack with O'Keefe's. This has taken the form of a Printpack study to establish a precision and accuracy level for the technique, to reduce the signal to noise ratio, and to confirm a limit of detection. *These efforts to refine the chromatographic method have been successful in all aspects: precision and accuracy levels, limits of detection, and signal-to-noise ratio.*

4. Tasks 4,6,7 (MWS process)

Review of the previous US FDA validation of the WSU MWS process for mashed potatoes revealed the need to confirm that the packaging material used to contain the product did not adulterate it by leaching migrating chemicals in to food. *Risk mitigation:* Printpack will provide single-sided migration studies of the pouch materials used in last year's and this year's MWS chicken and dumplings. This experimental protocol will most likely conform to the US FDA "Chemistry Guidelines" for its "Food Contact Material Notification" program, but is subject to discussion with and concurrence by US FDA process authorities. *WSU has provided "MWS-treated samples" of last year's pouch material to compare with control stock, using Covance Laboratory, Madison, Wisconsin to provide the migration studies.*

The all-plastic laminated material used to make the pouches for last year's Chicken and Dumpling entrée presented a spotted pattern of small voids visible from the outside of the pouches. While not affecting the functionality of the material, the effect was definitely an unacceptable defect that is not acceptable in the future. *Risk mitigation:* Printpack has initiated a statistical analysis of laminator operating conditions intended to isolate the critical variables responsible for the defect. Additionally, mechanical modifications have been designed to widen this window of operability. These will be installed in January, 2010 at which time another statistical analysis will confirm process capability. *Analysis of standard operating conditions with the new mechanical improvements now indicates that we have a sustainable solution to this issue.*

The WSU MWS process for mashed potatoes defined an acceptable method for inoculating the product with appropriately thermal resistant-spores subject only to placement at the cold spot of the tray/lidding package. While identifying the cold spot in a pouch is a similar process, each of the elements of the diverse chicken and dumpling food product has its distinct thermal heating properties. MWS process validation requires: convincing assessment of the combination of food component/pouch position that is least susceptible to heat sterilization; spore inoculation of that position, and incubation of pouches to determine if those spores survived a given thermal treatment. *Risk mitigation:* WSU personnel have already analyzed the pouches used to contain last year's MRE entrees to identify the cold spot in a homogenous product. They have also assembled thermal

data on the product's components and have now determined that the dumplings represent the least thermally susceptible component, as a consequence of both their size and thermal properties. *With this early identification of critical process validation elements, they are developing inoculation techniques that will satisfy US FDA requirements.*

5. Tasks 8,9 (Shelf life Modeling)

   The M-Rule Container Performance Model for Foods has been developed and validated foods and packaging materials on less complex than those involved in shelf stable combat rations. Its adoption to these systems cannot be claimed to forecast shelf life performance until experimental data is developed for comparison to predicted results. *Risk mitigation: Printpack will use the model to validate input variables for the advanced barrier films by "modeling" the base films with standard laboratory assumptions of temperature, pressure, and humidity.*

6. Tasks 10,11 (TTI Technology)

   The time temperature indicator system used to print an indicator message on packaging material must resist not only premature development of indicia during package material conversion, material shipment and storage, product packaging and sealing but also early message fade during post-processing shipment and storage. *Risk mitigation:* Printpack and Segan have agreed to evaluate three Segan chemistries in the label (Task 10) phase. The WSU thermal cell in oil bath technique will be adapted to quantify and compare the color change kinetics of the three chemistries. This information will be used to select a chemistry to be adapted to a thermally resistant flexographic ink used to print laminations used to make pouches.

Program Risks

7. Task 1 (Laminations): (None…this task is effectively complete)
8. Task 2 (Physical, Barrier, Optical Data): As we found last year, OTR and WVTR measurement for the high barrier laminations is the rate limiting work step for this task. *The projected time required to obtain all OTR and WCTR measurements, indicates that we need to outsource the remaining testing as necessary to complete the data by the end of June.*
9. Task 3 (Photodegradation Data): With the positive results to date in transferring the Virginia Tech methodology to the Printpack lab, *we recognize little to no program risk for this task.*
10. Tasks 4,5,6,7 (Packaged Products): Timely completion of this task is contingent on coordination of the availability of Printpack's packaging material with product (entrée, sauce, and dessert bar) processing. In the former area, we have advised suppliers of the expected optimum raw materials of our planned orders for delivery in late spring and are confident of timely production and availability of the laminations. For the latter area, we would expect to utilize the same military contractors (Thermo Pac and Sterling Foods) used in the previous year's program.

Risks

Entree production is contingent on WSU resources. We expect regular, at least weekly communication with WSU staff regarding plans and scheduling of entrée preparation, packaging, and processing. *WSU administration has committed to maintain the WSU MWS process pilot plant at its present location through the end of December, 2010. Additionally, Printpack plans to participate in the commercially-oriented Microwave Consortium being organized to scale up the technology from this pilot scale.*

11. Tasks 8 & 9 (Shelf life Modeling): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.
12. Tasks 10 &11 (TTl Technology): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.

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Issues

Unexpected Issues

TASK AREA	COMMENT
1. Task 1 (Laminations):	(none)
2. Task 2 (Barrier data):	• The very low transmission rates, both OTR and WVTR) of the test laminations has delayed completion of the Task 2 deliverable. Already over 3500 hours of instrument time have been consumed to produce about 40% of the required data. Barrier measurement will be outsourced to complete this task by the end of June.
3. Task 3 (Photodegradation Data)	(none)
4. Tasks 4,6,7 (MWS process)	• The need for data confirming that the MWS process itself does not cause chemical changes in packaging materials that are otherwise compliant with US FDA requirements for high temperature sterilization was not recognized in the original project scope (or original FDA validation) Directed studies to provide such data (according to US FDA protocols*) are available through industry laboratories (Cost estimates have been requested) • A whole muscle item (salmon with Alfredo sauce has been added as a second FDA process validation subject. WSU has not yet been able to secure an appropriate supply of salmon for the MRE portion size.
5. Tasks 8,9 (Shelf life Modeling)	(none)
6. Tasks 10,11 (TTI Technology)	• Negotiating acceptable subcontract delayed the start of this task, but the rapid WSU test cell evaluation method will accelerate the deselection phase of this work.

* http://www.fda.gov/Food/GuidanceComplianceRegulatoryInformation/GuidanceDocuments/FoodIngredientsandPackaging/ucm081818.htm. See especially "APPENDIX II. SELECTED MIGRATION TESTING PROTOCOLS"

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Details

Good News

TASK AREA	COMMENT
1. Task 1 (Laminations):	▪ New improved WVTR films from Toppan (GL-ARHF) provided expected improved results.
2. Task 2 (Barrier data): 3. Task 3 (Photodegradation Data)	▪ Photodegradation data indicates that 2 layers of pigmented adhesive is sufficient to protect lipids from photooxidation
4. Tasks 4,6,7 (MWS process)	▪ The WSU MWS pilot line will be available for trials throughout all of 2010.
5. Tasks 10,11 (TTI Technology)	▪ Work has begun. Testing in WSU oil bath cell calibration method will accelerate initial system development and confirmation of functionality

(ToC)

Details

Technical

TASK AREA	COMMENT
6. Task 1 (Laminations):	▪ Laminations complete and submitted. Detailed listing in Annex A
7. Task 2 (Barrier data):	▪ Available barrier data is summarized in Annex B.
8. Task 3 (Photodegradation Data)	▪ Photodegradation Data complete. (Annex C). ▪ Other physical data is complete for the 10 laminations (Annex D) ▪ WSU has completed its thin film MW resonance testing on the 10 laminations (Annex E).
9. Tasks 4,6,7 (MWS process)	▪ The WSU MWS process has received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding. ▪ Printpack has completed a designed experiment with its commercial intended to identify the root cause(s) of the "pocked" appearance of laminations made last year. (Annex E)
10. Tasks 8,9 (Shelf life Modeling)	▪ Work has begun.
11. Tasks 10,11 (TTI Technology)	▪ Confirmed the ability to test the performance of the TTI technologies (at the label stage) with the WSU Test Cell technique, allowing rapid screening of options and potentially development of a full kinetics description of the TTI color change.

(ToC)

Financial

Project Expenses as of 26 Mar 2010

COST ELEMENT	Contract Amt	Qtr 1 Amt	Qtr 2 Amt
Total Direct Labor	232,930	28,496	24,112
Payroll Tax & Benefits	84,511	10,339	8,748
Departmental Overhead	135,996	16,637	14,077
Labor Total	453,437	55,472	46,938
Consulting & Services	544,500	0	941
Materials	70,850	33,170	-
Travel	24,530	6,660	4,152
Other Direct Costs	160,760	99,505	*(6,807)
Subtotal Costs	1,254,077	194,807	45,224

** Adjustment for FY'08 Costs improperly charged to FY'09 Project.*

Project expenses to date as captured in Printpack's financial accounting system indicate that the work remains on budget. The relatively low expense amount to date reflects the lack of any payments made to the two expected subcontractors and captured in the system. However, in both cases, subcontract work has begun, and the record of payments will reflect this work and more by the next quarterly report

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Equipment

Equipment*

Equipment	Cost	Assoc. Task(s)	Location
OXTRAN® 2/21 SL Env. Chamber Operating. System	$61,641	2	Printpack Analytical Services Lab 5 Barber Industrial Ct. Villa Rica, GA 30180
Leap Autosampler System	$36,107.	3	

* No addirional equipment purchases during 2nd quarter

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Subcontracts

Subcontractor	Cost	Assoc. Task(s)	Status
Washington State University	$400,533	4,6,7	Signed; work underway
Segan Industries	$144,000.	10.11	Signed; work underway

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Annex A: Announcement of USFDA Validation of WSU MWS Process

FDA Approves WSU Researcher's Revolutionary New Food Processing Technology

Wednesday, Oct. 28, 2009

http://www.wsunews.wsu.edu/pages/publications.asp?Action=Detail&PublicationID=16596

PULLMAN, Wash. - Imagine a salmon filet that looks, tastes and is as nutritious as freshly cooked salmon but has a shelf-life of more than six months. A new technology developed at Washington State University will make that dream a reality.

For the first time ever, the U.S. Food and Drug Administration has approved the use of microwave energy for producing pre-packaged, low-acid foods, a major milestone that clears the way for its commercialization. The technology developed at WSU could revolutionize how we preserve and process food.

Juming Tang, a professor in the WSU Department of Biological Systems Engineering, led a team of university, industry and U.S. military scientists to develop the technology. The outcome results in food with a longer shelf life as well as better flavor and nutritional value compared to more traditional food processing methods such as canning.

"New processes for producing shelf-stable, low-acid foods must pass rigorous reviews by FDA to ensure that the technology is scientifically sound and the products will be safe," Tang said. "Our team patented system designs in October 2006 after more than 10 years of research. We spent another three years, developing a semi-continuous system, collecting engineering data and microbiologically validating the process before receiving FDA acceptance."

The team's Microwave Sterilization Process technology immerses the packaged food in pressurized hot water while simultaneously heating it with microwaves at a frequency of 915 MHz — a frequency which penetrates food more deeply than the 2450 MHz used in home microwave ovens. This combination eliminates food pathogens and spoilage microorganisms in just five to eight minutes and produces safe foods with much higher quality than conventionally processed ready-to-eat products.

Spearheaded by C. Patrick Dunne, Department of Defense combat feeding directorate at the U.S. Army Soldier Systems Center at Natick, Mass., the project has been funded from a variety of sources and a consortium of industry members that include Kraft Foods, Hormel, Ocean Beauty Seafoods, Rexam Containers, Ferrite Components and Graphic Packaging. The WSU team also worked closely with process authorities of the Seafood Products Association in Seattle and Hormel to establish validation procedures and in preparation of filing documents. In addition, faculty members from other WSU departments, particularly Food Science, contributed to the success of the project.

"The team's collective efforts have brought a new food processing technology to the forefront which will truly benefit not only the commercial sector but our war-fighters worldwide with a wider variety of high quality, shelf-stable foods," said Gerald Darsch, director of the U.S. Department of Defense Combat Feeding team. "It is truly a tremendous accomplishment."

Evan Turek, senior research fellow at Kraft Foods, said Tang's new technology will make a huge difference for the food industry.

"Since the introduction of industrial microwave ovens in the late 1940s, the food industry has been interested in exploiting the rapid heating capability of microwaves to

improve the quality of canned food," he said. "The technical issue has always been ensuring uniform and reproducible heat treatment. Dr. Tang had a vision about how this might be overcome, and with his leadership and the engineering prowess of his research staff and students, a protocol for practicing and validating microwave sterilization was established. Kraft Foods is proud to have been an early supporter of the research program at WSU and looks forward to the commercialization of the technology."

WSU officials agreed.

"This is a great example of how research universities like Washington State University produce breakthroughs that make an immediate impact on our nation and world. This new technology promises significant advances in food safety and quality to benefit everyone," said Howard Grimes, vice president for research.

Dan Bernardo, dean of the WSU College of Agricultural, Human, and Natural Resource Sciences, said the impact of the science will be dramatic.

"There have been very few advances leading to FDA accepted food processing technologies in recent history," he said. "The FDA's approval of this new technology truly could revolutionize the way we process and preserve food, ensuring food safety, increasing its longevity and maximizing the retention of its flavor and nutrition."

Ralph Cavalieri, director of the WSU Agricultural Research Center, said Tang's research has global benefits.

"It is important across a range of applications," he said, "from feeding astronauts on long-term space missions or soldiers in the field to transporting and storing food to areas of the world where people are unable to produce enough food locally to feed themselves."

Cavalieri said the project would not have been possible without support from a variety of sectors.

"We have worked synchronously with industry, the army and the university to make this happen," he said. "Dr. Tang's research also has received incredible support from Washington's Congressional delegation, especially Sen. Patty Murray."

Sen. Murray said ensuring funding for projects such as Tang's is part of an overall effort to support Washington's agricultural and food industry in ways that benefit the nation and world.

"This is great news for WSU, our growers and American food processors," she said. "It will help our growers and processors stay more competitive in the global marketplace and increase food safety for consumers

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Annex B: Working Specification for TTI

TTI Design Criteria: Microwave Thermal Sterilization Process

Print/Processing Conditions	Duration	Exposure	State Change	Pressure
Packaging Material				
Flexographic print/drying:	less than 1 second	80 – 100°C	reversible	Ambient
Nip press heat lamination:	less than 1 second	80 – 90°C	reversible	20-60 psi
Storage stability:	3-6 Months	20 – 25°C	reversible	Ambient
Product & Processing				
Hot filling	10 – 30 minutes	70 – 90 °C	reversible	Ambient
Pre warming	10 – 30 minutes	70 – 100°C	reversible	Ambient
Microwave sterilization:	3-5 Min.	120 – 130°C	irreversible	30 psig
Post processing storage	2 years	0 – 60°C	no reversion	Ambient

MTSP Sensing Composition Criteria: Working Criteria

- Compatible with flexographic printing on oriented Polyester film: solvent or aqueous based without use of "hazardous air pollutants" *http://www.epa.gov/ttn/atw/188polls.html*
- Survive reversible conditions up to 100°C and becomes irreversible above 120°C
- Must survive pressure, lamination step, and chemical interaction with laminate
- Visually addressable after complete cycling and readable after package cools
- Microwave compatible – responds to conductive heat from heated product not microwave energy, no metal allowed
- Must not revert during post storage conditions of 3 years at 27°C (80°F) or 6 months at 38°C (100°F) [i.e. until consumption]

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Annex C: WSU Subcontract Budget

Juming Teng application to Springpack
July 1, 2010 - June 30, 2011
John Anderson
335-6642; fax 335-2722
jra@wsu.edu

PI's Name:						Year 1
Agency:						
SALARIES - 01	**Pay Role**	**# Mos.**	**% FTE**			
Fang Liu	Personal & Confidential				**Salary**	29,151
				Benefits	32.00%	9,328
Galina Mikhaylenko					**Salary**	17,879
				Benefits	42.00%	7,509
Jae Hyung Meh					**Salary**	19,470
				Benefits	43.00%	8,372
Zhongwei Tang					**Salary**	20,520
				Benefits	41.00%	8,413
Juming Teng					**Salary**	16,508
				Benefits	29.40%	4,853
PhD Student			1		**Salary**	14,734
					QTR	8,105
					Health	1,613
					1.5%	221
Master Student			0		**Salary**	-
					QTR	-
					Health	-
					1.5%	-
WAGES - 01	**$ Per Hr.**	**Hrs/Wks**	**# Wks.**			
Student (full-time):	$0.00	0	0		**Wages**	-
				Benefits	2.1%	-
Student (part-time):	$20.46	20	13		**Wages**	5,320
(graduate student in summer)				*Benefits*	9.7%	516
					Total Salary	118,262
					Total Wages	5,320
					Total Salary & Wages	123,582
BENEFITS - 07						
					Total Benefits	48,930
					Total Salaries/Wages/Benefits	172,512
CAPITAL EQUIPMENT - 06 ($5,000 +)						
Residue gas measurement device						8,000
Network Analyzer						37,000
Single-mode resonance cavity for package material dielectric property measurement						7,000
					Total Capital Equipment	45,000
GOODS/SERVICES - 03						
Rental for FCI generator, directional couplers, waveguide						57,500
Materials and chemicals						4,000
Fiber optic sensors, Ellab sensors, replacement parts						3,000
Off-Site Rental						
Non-Capital Equipment (Under $4,999)						
Other						
					Total Goods/Services	64,500
TRAVEL - 04						
Domestic						3,500
Foreign						
					Total Travel	3,500
TOTAL DIRECT COSTS						285,512
EXCLUSIONS						
QTR						8,105
Equipment (Over 5k)						45,000
Subcontracts (After Initial $25K For Each Subcontract)						-
Other (Off-Site Rental & Stipends, Etc)						
					Base	232,407
			F&A Rate	**MTDC**	49.5%	115,041
			F&A Rate	**Total Cost**	0.0%	-
			F&A Rate	**Total Dir**	0.0%	-
TOTAL F&A - 13						115,041
TOTAL COSTS						400,553

F&A Base Type:	MTDC	TD	TC	SWB	Other
	X				

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Annex D: 10 Barrier Laminations

Lam Number	Outer Layer	Barrier Layer	barrier2 Layer	Sealant Layer	COMMENTS
1*	OPET	KUR-C	KUR-N	Cblack B343T-997	Carbon Black Sealant w/ opaque adhesive
2*	OPET	KUR-C	KUR-N	CMYW B343T-997	CMYW Black Sealant w/ opaque adhesive
3*	OPET	KUR-C	KUR-N	Clear B343T-997	Clear Sealant w/ 2 opaque adhesives
4*	OPET	KUR-C	KUR-N	Cblack B343T-997	Carbon Black Sealant
5#	OPET	GL-PET-ARH	KUR-N	Clear B343T-997	Retort Grade GL OPET
6#	OPET	GL-PET-ARHF	KUR-N	Clear B343T-997	Sub-Retort Retort Grade GL OPET
7#	OPET	SX03 Y07	KUR-N	Clear B343T-997	SKC PvDC_Ctd Barrier OPET
8^	OPET	GL-PET-ARHF	BON	Clear B343T-997	GL Barrier only
9^	OPET	KUR-C	BON	Clear B343T-997	Kurarister-C Barrier 4-ply lamination
10^	OPET	KUR-C	n/a	Clear B343T-997	Kurarister-C Barrier 3-ply lamination

*: Alternate opaque sealant.
#: Control and better WVTR
^: Lower cost alternatives

Adhesive
Clear
Pigmented

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Annex E: DOE for Buckling Reduction

Buckling is defined as web wrinkling in the cross direction (CD) of a web. The causes of buckling are great and varied including defective raw material (baggy, wrinkled, etc), machine misalignment and air entrapment at the rewind. In this Design of Experiment (DOE) we are looking at factors that we have the most control of in our converting facility, namely air entrapment at the rewind (machine alignment will be addressed with installation of the outfeed nip). The four main inputs in rewinding were selected as variables for the experiment.. We performed two sets of experiments utilizing both fiber and steel cores to cover the wide range of structures and machine conditions that we run.

Minitab was used to both design the experiment and analyze the data. A 2-level factorial DOE was used with 4 variables, resulting in 9 sets of run conditions for each type of core. This balanced both practicality of the trial with sufficient resolution to obtain statistically significant results. Trials were run on consecutive days holding all machine conditions constant including staffing to minimize variability. Data was gathered watching each roll as it was unwound on the slitters. Buckling severity was gauged and recorded for each master roll, noting both extent and severity of buckles. A scale was created from 0 – 5 with 0 being no buckling observed to 5 being severe buckling from core to top of roll and extending across the face of the roll (See Appendix A).

The Main Effects plots (Appendix B) from Minitab show the individual contributions of each factor to buckling. These plots seem to mirror each other, except in the Variable D plot. Upon further research and analysis, I found two distinct factors that contributed to the differences: taping of cores and machine capability. First, the fiber cores were taped in a spiral direction that at certain conditions failed to adhere to the core and caused buckling from uneven transfer of the web. Also, the machine is not capable of running the full range of settings for variable D. Conclusions were made that the greatest factor influencing buckling is variable A, followed by Variable B, to some extent variable C and lastly, variable D.

The Interaction plots show the most effective combinations of each factor in creating or reducing buckling. These plots showed the dominance of Variable B over all other factors in regards to buckling. This is very evident in the steel core trials where the machine's limit for variable B was applied. The results are conclusive that maximizing variable A and variable B were most effective in reducing buckling.

One factor that was found to be significant during this trial, yet not relevant to machine conditions is the raw material. We witnessed varying degrees of buckling that could be directly correlated to the profile of the sealant film we were running. Baggy edges or lanes were prone to buckling, especially on low tension runs. We were however able to minimize the effects of the profile issues with certain process conditions..

In general, increasing variable A and variable B are the most effective means of reducing buckling. To a lesser extent, decreasing machine variable C can also have a positive impact on buckling, but as a last resort when maximum feasible levels for variable A and variable B are reached for any given structure. While these results do show us the means by which we can reduce buckling, they do not give us the exact settings for every structure to eliminate buckling. By utilizing our Standard Operating Conditions

(SOC's) and applying these findings we should be able to minimize buckling on the adhesive laminator and other similar pieces of equipment.

Appendix A – Parameters and Results

Fiber Core:

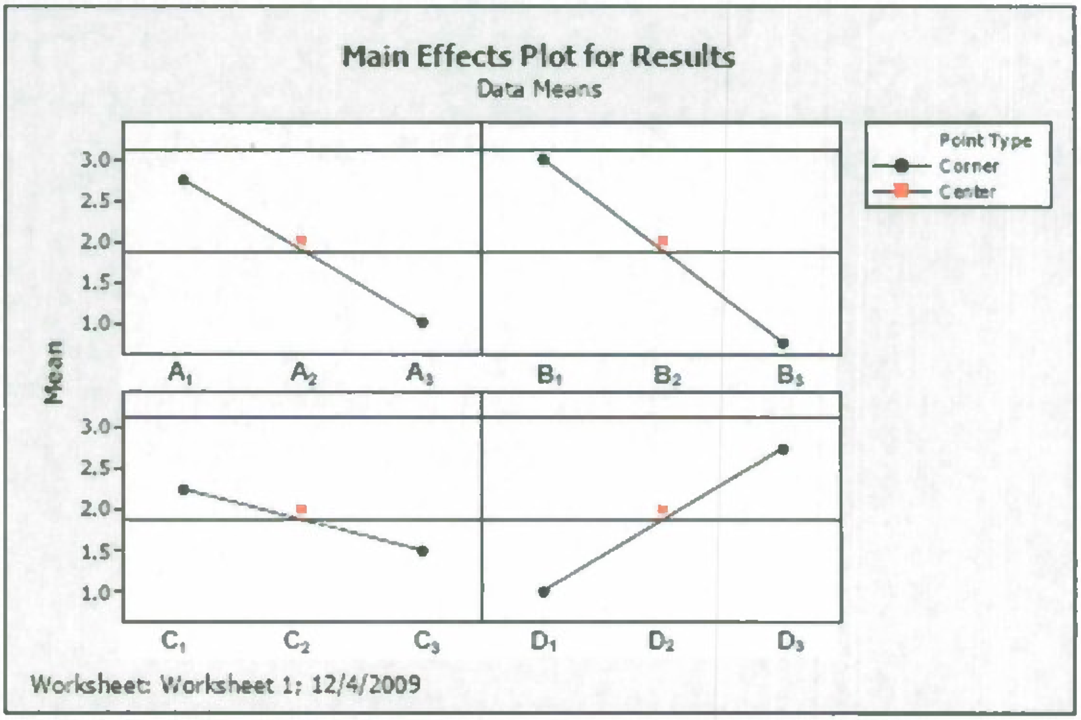
Run Order	Variable A	Variable B	Variable D	Variable C	Results
1	1	1	1	1	3
2	3	3	3	3	0
3	2	2	2	2	2
4	3	1	3	1	0
5	1	3	1	3	2
6	3	3	1	1	0
7	1	3	3	1	1
8	3	1	1	3	4
9	1	1	3	3	5

Fiber Core:

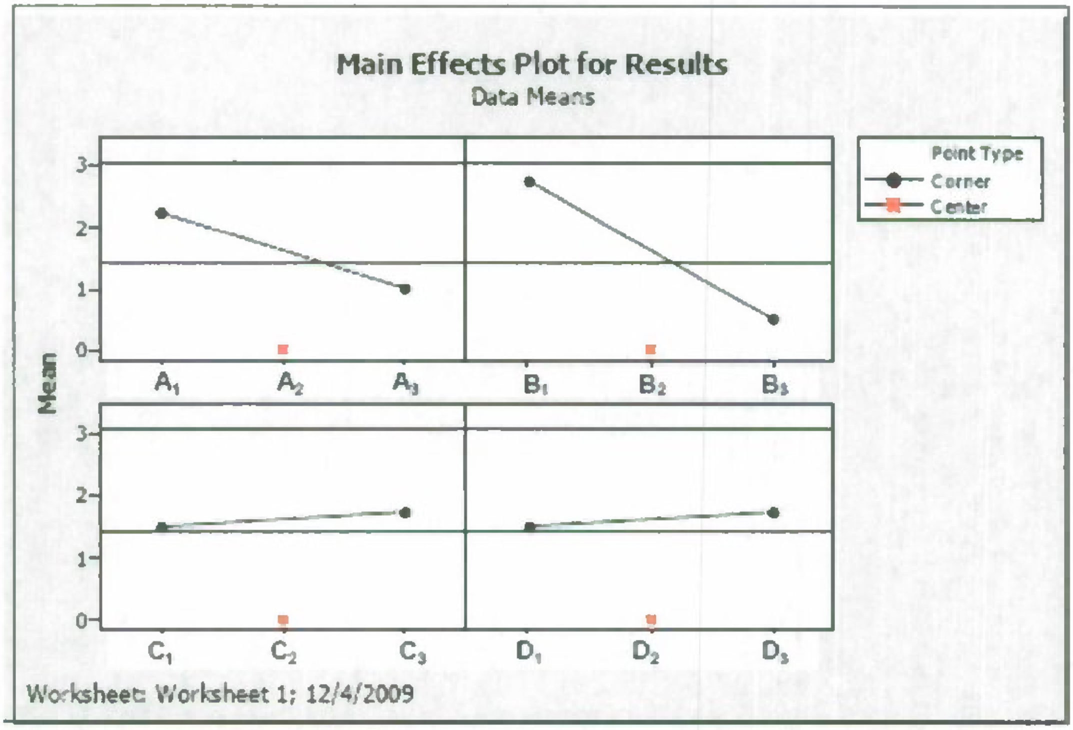
Run Order	Variable A	Variable B	Variable D	Variable C	Results
1	1	1	1	1	3
2	6	3	3	3	0
3	2	2	2	2	0
4	6	1	3	1	2
5	1	3	1	3	1
6	6	3	1	1	0
7	1	3	3	1	1
8	6	1	1	3	2
9	1	1	3	3	4

Appendix B – Main Effects Plots

Fiber Core

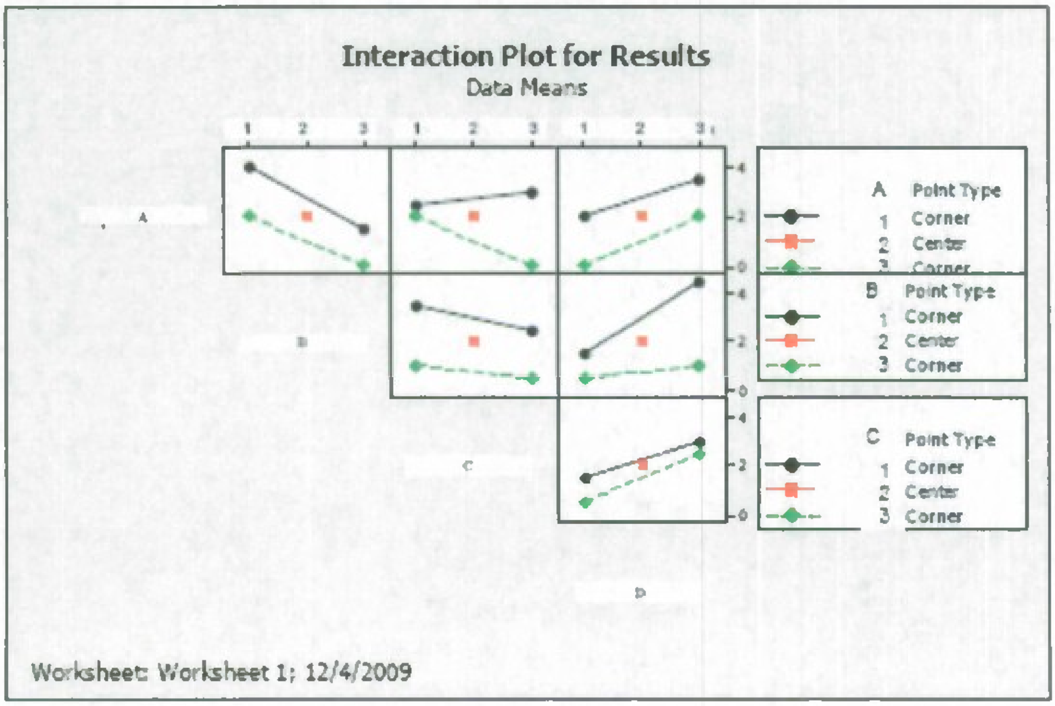


Steel Core

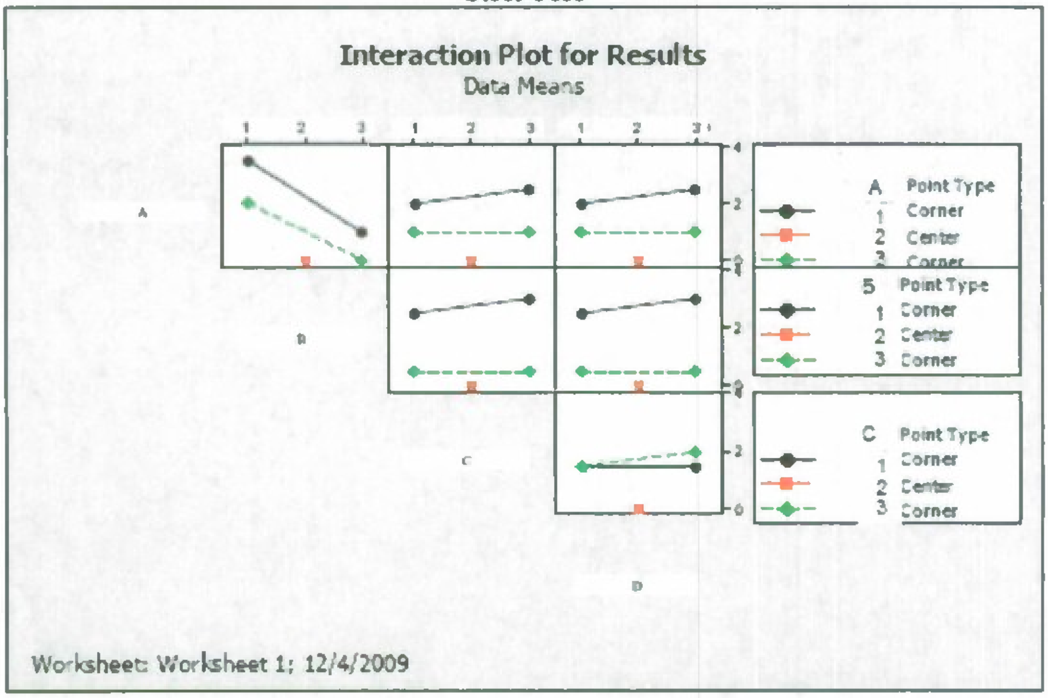


Appendix C – Interaction Plots

Fiber Core



Steel Core



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ANNEX F: SPME-GC-MS Method

Gas Chromatograph: HP-Agilent 6890N

- Column: Agilent Carbowax 20M, 30 x 0.25 x 0.25
- Carrier gas: Helium 1.5 ml / min, constant flow mode
- Inlet temp: 270 °C
- Injection mode: splitless
- Oven:
  1. 40°C (1 min hold)
  2. 5°C gradient / per min to 200°C (hold 2 min)
- Auxiliary line to MSD: 210°C

Detector: HP-Agilent 5973 Mass Selective Detector (MSD)

- Scan range *m/z* 50-550
- Autosampler: LEAP Technologies CombiPal

SPME fiber: DVB/CAR/PDMS

- Extraction time: 15 min with agitation
- Extraction temp: 60°C

Validation to include:

- linear dynamic range,
- precision,
- accuracy,
- recovery (function of the amount of yogurt added to vial),
- specificity

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Quarterly Report

For the Period Ending
30 June, 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Quarterly Report

For the period ending
30 June 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Summary: After two quarter, the project is on budget with slippage in some early tasks. The deliverables 1 and 3 have been submitted. Significant progress for deliverables 2 and 4 through 7 is reported here.

Project Overview

Objective

To advance the state of the art for Thermal Microwave Sterilization (MWS) for pouches of individual combat rations while maintaining current shelf life:

- improvements in the MWS process at Washington State University (WSU) and
- improvements in the non-foil light barrier materials considered in the Print-pack research

Deliverables

Task	Deliverable	Plan date	Act/Ant date
1	10 Laminations	31 Dec 2009	08 Jan 2010
2	Physical, Barrier, & Optical Data	28 Feb 2010	31 Aug 2010
3	Photodegradation Data	31 Jan 2010	5 Mar 2010
4	Retort & MWS Entrée Packages	30 Apr 2010	TBD
5	Hot Fill Packages	30 Apr 2010	31 Aug 2010
6	Optimized MWS Entrée Packages	30 Jun 2010	TBD
7	MWS Validation Report	30 Jun 2010	TBD
8	Standard Condition Shelflife Modeling	30 Apr 2010	31 Aug 2010
9	Extreme Condition Shelflife Modeling	31 May 2010	31 Aug 2010
10	TTI Label Evaluation	30 Apr 2010	1 July 2010
11	Printed Pouch TTI Evaluation	31 Aug 2010	31 Aug 2010

(ToC)

Accomplishments

1. Task 1 (Laminations): All laminations are completed; including advances include new barrier materials and alternate opacifying pigments for sealant films. (Details provided in "ANNEX A" from March, 2010 Report.)
2. Task 2 (Material data sheets): High barrier performance of materials results in lengthy cycle times for each measurement of OTR and WVTR. Only these data remain for completing this task (Available Data in ANNEX A.) The thin film dielectric property measurement technique developed at WSU during last's year's project continues to provide useful guidance about the materials' energy interactions in MWS. Values for each lamination made in Task1 have been developed with this method an incorporated into data sheets.
3. Task 3 (Photodegradation Data): the Virginia Tech photodegradation assessment technique has been transferred to Printpack's Analytical Laboratory. Validation of the technique and calibration to the previous year's Virginia Tech results proved difficult because experimentally detected levels are at or below the limits of detection. Dr. Sean O'Keefe of the Virginia Tech Food Science Department, author of the previous year's report, has been very helpful and supportive of this effort. (Report previously submitted)
4. Tasks 4, 6, 7 (MWS process): Professor Juming Tang of Washington State University (WSU) has requested a 12-month co-cost extension of this subcontract. Details of proposed revision are provided in "Subcontracts" section. Discussions currently in progress to achieve submission of an FDA validation report in the first quarter of CY2011 now underway.
5. Tasks 8 &9 (Shelf life Modeling): Validation of loaded data continues to take longer than anticipated for both the initial phase (standard distribution.) and the second phase (extreme distribution systems).
6. Tasks 10 & 11 (TTI Technology): Segan Industries (Burlingame, CA) subcontract for the development of a flexographic ink providing Time Temperature Indications on microwave sterilized pouches has proceeded satisfactorily. "Immediate solutions" have been developed and tested (see ANNEX C for preliminary draft). Intermediate solutions have already been evaluated in Printpack's temperature-resistant ink vehicle and plans for press trial and MWS evaluation are under way.

(ToC)

Risks

Technical and program Risks

Technical Risks

1. Task 1 (Laminations): (None… task effectively complete)
2. Task 2 (Physical, Barrier, Optical data):

   Two novel barrier films are added to the set used in the laminations from last year's project. Both provide improved water vapor barrier transmission rate (WVTR), but while compliant with US FDA requirements for high temperature cook-in processes, they are not intended for use at 121°C for the duration of a full retort cycle. *Risk mitigation:* To assess the thermal stability of laminations with these films, we will add WVTR and oxygen transmission rate (OTR) *following thermal abuse* to the flat and mechanically-abused barrier data provided for the other laminations. Filled Pouches will be processed at 121°C in the Clemson University Packaging Science Department's retort chamber. We will use a 40 minute retort cycle and a 7 minute cycle to imitate the MWS process. Material from the treated pouches will be statistically analyzed to determine if WVTR and OTR are significantly affected. (Printpack's membership in the Clemson Center for Excellence in Flexible packaging- CEFPACK- will underwrite these studies). *This effort will be initiated when the current backlog of barrier testing is relieved.*

3. Task 3 (Photodegradation Data) : (None… task effectively complete)

4. Tasks 4,6,7 (MWS process)

   Review of the previous US FDA validation of the WSU MWS process for mashed potatoes revealed the need to confirm that the packaging material used to contain the product did not adulterate it by leaching migrating chemicals in to food. *Risk mitigation:* Printpack will provide single-sided migration studies of the pouch materials used in last year's and this year's MWS chicken and dumplings. This experimental protocol will most likely conform to the US FDA "Chemistry Guidelines" for its "Food Contact Material Notification" program (see "Issues" summary for reference), but is subject to discussion with and concurrence by US FDA process authorities. *WSU has provided "MWS-treated samples" of last year's pouch material to compare with control stock. Discussions with Covance Laboratory, Madison, Wisconsin to provide control materials is under discussion.*

   The WSU MWS process for mashed potatoes defined an acceptable method for inoculating the product with appropriately thermal resistant-spores subject only to placement at the cold spot of the tray/lidding package. While identifying the cold spot in a pouch of heterogeneous components is a similar process, each of the elements of the diverse chicken and dumpling food product has its distinct thermal heating properties. MWS process validation requires: identifying the combination of food component and pouch position that is least susceptible to heat sterilization; spore

inoculation of that position, and incubation of pouches to determine if those spores survived a given thermal treatment. *Risk mitigation:* WSU personnel have already analyzed the pouches used to contain last year's MRE entrees to identify the cold spot in a homogenous product. They have also assembled thermal data on the product's components and have now determined that the dumplings represent the least thermally susceptible component, as a consequence of both their size and thermal properties. *With this ongoing work to identify critical process validation elements, they are developing inoculation techniques that will satisfy US FDA requirements.*

5. Tasks 8,9 (Shelf life Modeling)

   The M-Rule Container Performance Model for Foods has been developed and validated foods and packaging materials on less complex than those involved in shelf stable combat rations. Its adoption to these systems cannot be claimed to forecast shelf life performance until experimental data is developed for comparison to predicted results. *Risk mitigation:* Printpack will consult with the Model's developer to adapt its input requirements to these complex non-foil barrier laminations.

6. Tasks 10,11 (TTI Technology)

   TASK 10 (Immediate Solutions): (None… task effectively complete)

   TASK 11 (Intermediate Solutions)

   The time temperature indicator system used to print an indicator message on packaging material must resist not only premature development of indicia during package material conversion, material shipment and storage, product packaging and sealing but also early message fade during post-processing shipment and storage. The formulation with its TTI functionality must be acceptable in Printpack's printing presses. *Risk mitigation:* Printpack and Segan complete the evaluation of three Segan chemistries in label form in WSU pilot plant runs. This information will be used to select a chemistry to be adapted to a thermally resistant flexographic ink used to print laminations used to make pouches. Segan prepared these chemistries in Printpack's thermally-resistant ink vehicle. To anticipate the printability of this formulation must be acceptable in a commercial printing process: 1) Printpack will supply a small role of 12 μ OPET film for bench top evaluations of the following samples; 2) A sample of a press ready Printpack ink will be provided and the Segan formulation's viscosity matched to it; 3) A sample of Printpack's pigmented adhesives system will be provided and the Segan formulation's functionality evaluated

Program Risks

7. Task 1 (Laminations): (None…this task is effectively complete)
8. Task 2 (Physical, Barrier, Optical Data): As we found last year, OTR and WVTR measurement for the high barrier laminations is the rate limiting work step for this

task. *The projected time required to obtain all OTR and WCTR measurements, indicates that we need to outsource the remaining testing as necessary to complete the data by the end of June.*

9. Task 3 (Photodegradation Data): (None…this task is effectively complete)
10. Tasks 4,5,6,7 (Packaged Products): Printpack's packaging material is now available, but WSU faculty and staff have been focused on developing and submitting documentation for a salmon in Alfredo sauce product. Inoculated pouches of this product are now undergoing incubation. *WSU administration has committed to maintain the WSU MWS process pilot plant at its present location through the end of December, 2010, but they have requested a no-cost contract extension through the end of FY2011. Discussions are now underway to*
11. Tasks 8 & 9 (Shelf life Modeling): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.
12. Tasks 10 &11 (TTI Technology):

    TASK 10 (Immediate Solutions): (None… task effectively complete)

    TASK 11 (Intermediate Solutions): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.

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Issues

Unexpected Issues

TASK AREA	COMMENT
1. Task 1 (Laminations):	(none)
2. Task 2 (Barrier data):	• The very low transmission rates, both OTR and WVTR) of the test laminations has delayed completion of the Task 2 deliverable. Already over 3500 hours of instrument time have been consumed to produce about 40% of the required data. Barrier measurement will be outsourced to complete this task by the end of June.
3. Task 3 (Photodegradation Data)	(none)
4. Tasks 4,6,7 (MWS process)	• The need for data confirming that the MWS process itself does not cause chemical changes in packaging materials that are otherwise compliant with US FDA requirements for high temperature sterilization was not recognized in the original project scope (or original FDA validation) Directed studies to provide such data (according to US FDA protocols*) are available through industry laboratories (Cost estimates have been requested) • A whole muscle item (salmon with Alfredo sauce has been added as a second FDA process validation subject. WSU has not yet been able to secure an appropriate supply of salmon for the MRE portion size.
5. Tasks 8,9 (Shelf life Modeling)	(none)
6. Tasks 10,11 (TTI Technology)	• Negotiating acceptable subcontract delayed the start of this task, but the rapid WSU test cell evaluation method will accelerate the deselection phase of this work.

* http://www.fda.gov/Food/GuidanceComplianceRegulatoryInformation/GuidanceDocuments/FoodIngredientsandPackaging/ucm081818.htm. See especially "APPENDIX II. SELECTED MIGRATION TESTING PROTOCOLS"

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Details

Good News

TASK AREA	COMMENT
1. Task 1 (Laminations):	▪ *New improved WVTR films from Toppan (GL-ARHF) provided expected improved results.**
2. Task 2 (Barrier data): 3. Task 3 (Photodegradation Data)	▪ *Photodegradation data indicates that 2 layers of pigmented adhesive is sufficient to protect lipids from photooxidation**
4. Tasks 4,6,7 (MWS process)	▪ *The WSU MWS pilot line will be available for trials throughout all of 2010.** ▪ Polymeric laminations for food processed produced with improved visual quality and full functionality. ▪ Pouches and roll stock are ready for processing trials.
5. Task 10 (Immediate TTI Technology)	▪ Work essentially complete and successful
6. Tasks 11 (Intermediate TTI Technology)	▪ Bench top testing of TTI pigment with standard flexographic heat-resistant vehicle confirmed functionality equal to or better than in previous screen printing vehicle

** Previously reported*

(ToC)

Details

Technical

TASK AREA	COMMENT
1. Task 1 (Laminations):	▪ Laminations complete (previously submitted).
2. Task 2 (Barrier data):	▪ Barrier data is summarized in ANNEX A.
3. Task 3 (Photodegradation Data)	▪ Photodegradation Data complete (previously submitted). ▪ Other physical data is complete for the 10 laminations (previously submitted) ▪ WSU has completed its thin film MW resonance testing on the 10 laminations (previously submitted)
4. Tasks 4,6,7 (MWS process)	▪ The WSU MWS process has received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding. ▪ Printpack has successfully laminated all polymeric high barrier structures for both shelf-stable thermally-processed food and hot fill items. WSU is behind schedule with developing engineering data and inoculation methodology.
5. Tasks 8,9 (Shelf life Modeling)	▪ Example data input computations provided in ANNEX B
7. Task 10 (Immediate TTI Technology)	▪ Preliminary report provided in ANNEX C
8. Tasks 11 (Intermediate TTI Technology)	▪ In depth evaluations of precise and repeated color indications finished on bench top and onto the WSU Pilot Plant for additional confirmation. ▪ Detailed preparations for press run are now in process at both Printpack and Segan.

(ToC)

Financial

Project Expenses as of 26 Mar 2010

COST ELEMENT	Contract Amt	Q-1 Amt	Q-2 Amt	Q-3 Amt
Total Direct Labor	232,930	28,496	24,112	34,396
Payroll Tax & Benefits	84,511	10,339	8,748	12,497
Departmental Overhead	135,996	16,637	14,077	20,081
Labor Total	453,437	55,472	46,938	66,974
Consulting & Services	544,500	0	941	87,909
Materials	70,850	33,170	-	16,153
Travel	24,530	6,660	4,152	11,705
Other Direct Costs	160,760	99,505	*(6,807)	19,144
Total Costs	1,254,077	194,807	45,224	201,858

** Adjustment for FY'08 Costs improperly charged to FY'09 Project.*

Project expenses to date as captured in Printpack's financial accounting system indicate that the work remains on budget. The increase in expenses for the 3rd quarter represents the initiation of the two subcontracts and production of the packaging materials for the food items. Much of the remaining (4th quarter) work will be conducted by the subcontractors. No budget problems are foreseen at this time.

(ToC)

Equipment

Equipment* ***(No change from last report)***

Equipment	Cost	Assoc. Task(s)	Location
OXTRAN® 2/21 SL Env. Chamber Operating. System	$61,641	2	Printpack Analytical Services Lab 5 Barber Industrial Ct. Villa Rica, GA 30180
Leap Autosampler System	$36,107.	3	

* No additional equipment purchases during 2nd or 3rd quarters

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Subcontracts

Subcontractor	Cost	Assoc. Task(s)	Status
Washington State University	$400,533	4,6,7	Signed; work underway*
Segan Industries	$144,000.	10.11	Signed; Phase 1 complete; work on Phase II underway

WSU Proposed Alternate Plan July 8 2010

STEP	ACTION	JUL	AUG	SEP	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG
1	Dielectric properties: Chicken & Dumplings	------	------	------	------										
2	Heat penetration Properties: Chicken & Dump-lings					------									
3	Technique for Spore Inoculation						------								
4	Process product for incubation study-preliminary							------	------	------					
5	Process product for incubation study-final										------				
6	Submit FDA Filing											------	------	------	
7	Response & additional data to FDA														------

WSU Original Work Plan July 8 2010

STEP	ACTION			OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP
2	Develop Thermal processing procedures: Chicken & Dumplings					------	------	------	------	------	------				
5	Process product for incubation study-final							------	------	------	------	------	------	------	------

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ANNEX A: Available Barrier data

	OTR, cc/100in²-day						MVTR, gm/100in²-day		
	OTR, dry, no flexes	OTR, dry, 5 Gelbo flexes	OTR, dry, 10 Gelbo flexes	OTR, wet, no flexes	OTR, wet, 5 Gelbo flexes	OTR, wet, 10 Gelbo flexes	MVTR, no flexes	MVTR, 5 Gelbo flexes	MVTR, 10 Gelbo flexes
Sample #21	<.0006	.0214/.0079	.0366/.0354				.2620/.0028	.2427/.0176	.2731/.0015
Sample #22	<.0006	.0119/.0010	.0124/.0008				.2738/.0008	.2557/.0245	.2687/.0004
Sample #23	<.0006	.0257/.0044	.0190/.0057				.2842/.0027	.2745/.0239	.2267/.0795
Sample #24	.0278/.0168	.2648/.0566	.2582/.0023				.2430/.0014	.2509/.0019	.1940/.0131
Sample #25	<.0006	.0204/.0086	.0128/.0075				.0222/.0003		.0384/.0002
Sample #26	<.0006	.0076/.0007	.0163/.0029				.0102/.0007		.0316/.0034
Sample #27	.0009/.0004	.0036/.0004	.2072/.0039				.2055/.0026		.0759/.0037
Sample #28	.0116/.0010	.1003/.0021	.0834/.0031				.0148/.0004		.0271/.0003
Sample #29	.0153/.0012		.1082/.0035				.2880/.0053		.2719/.0021
Sample #30	.0029/.0001		.0472/.0144				.2965/.0025		.2818/.0016

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ANNEX B

ANNEX B: Example data input computations (not complete as of 7/14/10)

Introduction

The M-RULE® Container Performance Model for Foods operates by integrating the fundamentals of permeant diffusion and solubility through polymeric materials, permeant vapor-liquid equilibriums, and time-dependent stress-relaxation behaviors with critically evaluated physical data for the component packaging materials. The model accommodates barrier coatings on standard polymeric materials with a user-supplied "Barrier Improvement Factor (BIF)". Instead of the diffusion and solubility appropriate for polymeric materials, the model calculates mass movement of permeant through such coatings by its inferring its flux through that coating.

Typical technical data available for flexible packaging films provides an oxygen transmission rate (OTR) for the material (typically "ASTM D3985 - 05 Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor") The rate is expressed in terms of the volume of oxygen (cubic centimeters) at standard conditions passing through a unit area (1 meter squared) of film with unit thickness[1] (25 microns) over a 24 hour period at specific temperature (°C), humidity (%RH) and partial pressure differential (atmosphere). This is a measure of the steady-state rate of transmission of oxygen gas through the polymeric material plastics. It provides for the determination of (*1*) oxygen gas transmission rate (OTR), (*2*) the permeance of the film to oxygen gas (PO2), and (*3*) oxygen permeability coefficient (P'O2) in the case of homogeneous materials. As such it is a contrived, laboratory benchmark useful for inter-material comparisons, but not an effective, predictive tool for shelf life prediction (unless temperature, humidity and the oxygen partial pressure differential for the packaged product are sustained as specified for the steady state testing). The M-RULE® handling of barrier coatings, simply assumes the mass transport through whatever voids exist in the coating as a function of the delivery of the permeant to the film/coating interface and the ability of whatever lies on the opposite face of the coating to remove the permeant (e.g. absorption by another polymer, dilution in a free atmosphere, etc).

Method

The oxygen solubility and diffusion of a uniform base film and the flux of oxygen through a coating on it are reflected in the OTR provided for a film coated with a particular barrier layer. Technical data on the OTR of base films and coated films are typically available. If attention is given to insure consistency of temperature, humidity, and partial pressure conditions, these data can be used to directly compute BIF values for the coated film:

$$BIF = \frac{OTR_{base}}{OTR_{ctd}}$$

Results

The coated films used in the sample laminations include only two base films: 15 μ biaxially-oriented Nylon-6 ("BON") and 12 μ biaxially-oriented polyethylene terephthalate

[1] In the case of coated film or multilayered materials, this assumption of uniform transport over a unit thickness is not appropriate and the OTR is reported per actual thickness.

(OPET). OTR values for these base films and the coated films used in the laminations are provided in Table 1. BIF values calculated from these data are also provided in the table…

Film	BIF
15m KUR-N	77.5
12μ KUR -C	77.5
12μ GL-AR-F	500
12μ 03-Y07	15.5

To verify these values, conditions for the model were programmed to reproduce the steady-state assumptions of ASTM D3985 for a model bag with 1 meter square surface area. The conditions were adjusted until the typical OTR rates for the base films (12μ OPET and 15μ OBON) were generated by the model. These same conditions were used with the indicated BIF for the coated films to model an OTR rates for comparison with their respective typical OTR rates.

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ANNEX C: Immediate TTI Technology

Development of Time/temperature Indicator Labels for Microwave Sterilization Process

Hans Ribi[2], Galina Mikhaylenko[3], Thomas Dunn[4]

Abstract

Co-topo-polymeric indicator compositions have been adapted as an ink medium suitable for confirming the exposure of a printed label on a flexible pouch to a target temperature for an indicated interval. The ink was printed onto heat resistant pressure sensitive-coated film and adhered to the outer surface (oriented polyester) of polymeric laminated pouches and processed in a microwave sterilization process. The observed color change confirmed the time/temperature exposure of the pouches in the process as confirmed by packaged electronic sensors.

Background

Military rations are currently packaged in multilayer aluminum-foil laminations which provide significant oxygen, water vapor, and light barrier. For a variety of reasons (Ratto et al., 2006) the military seeks to convert packaging for such rations to non-foil, polymeric packaging materials.

Microwave Sterilization (MWS) represents one major objective for replacing foil laminations (Tang et al. 2008). The process is a thermal one with the advantage of being able to raise pre-packaged contents of containers to sterilizing temperatures (121-125°C) rapidly (3-5 minutes) and maintaining target temperatures for the time required to kill pathogenic spores. This heating is quicker and degradation from heating is much less than in conventional retort processing (e.g. 20-30 minutes).

Operating and verifying the operation of a commercial MWS process requires reliable conformance to validated process conditions. Such conformance calls for sophisticated real time instrumentation of all parameters identified in the validation process. Reliable and accurate devices are of course crucial to this end. However, should such controls and backups fail, the shorter target time at sterilizing temperature for MWS implies that relatively small shortfalls will be more unsafe than in conventional thermal processes. An integral time/temperature indicator on the container will provide independent food safety and quality assurance for such contingencies.

As a step towards a heat resistant ink that can be printed on the packaging material, this research effort was devoted to evaluating the precision and accuracy of a model ink printed on pressure-sensitive labels and adhered to the outside of filled pouches.

The model ink is based on "Co-topo-polymers" disclosed in Ribi (2010). Compositions of these polymers are produced via polymerization of one or more monomeric components. The precursor compositions may have various ratios of distinct monomers, such as monomeric analogs, and may include one or more functional additives, e.g., that find use

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during co-crystallization. By way of example, consider the diacetylenic fatty acid, 2,4-Heneicosadiynoic acid (Cas No. 69288-33-1) [$CH_3(CH_2)_{15}C{\equiv}C{-}C{\equiv}C{-}COOH$]. The many areas of bond conjugation present in mixture of polymers including this and analogous diacetylenic monomers interact with light to produce color effects. Additionally, the inter-molecular rigidity resulting from multiple diacetylenic bonds in the polymer causes temperature dependence of the tertiary molecular and cystalline structure. This in turn changes interaction with light in reversible or irreversible ways.

Design latitude in time/temperature dependency of the pigment polymers results from selection of monomer hydrocarbon chain length (e.g., 10 to 30 carbon atoms long), head-group structure (e.g., ester, amide, etc.), bond positioning, appendages, chirality, related features, and/or combinations thereof. Table 1 summarizes the design specifications used for the composition developed here:

Table 1: Design Specifications for TTI label ink

Print/Process Conditions	Duration	Exposure	State Change	Pressure
Flexographic drying	< 1 sec.	80 – 100°C	reversible	Ambient
Heat laminating Nip	< 1 sec.	80 – 90°C	reversible	20-60 psi
Storage stability	3-6 Months	20 – 25°C	reversible	Ambient
Hot filling	10 – 30 min.	70 – 90 °C	reversible	Ambient
Pre warming	10 – 30 min.	70 – 100°C	reversible	Ambient
Microwave sterilization	3-5 Min	120 – 125°C	irreversible	30 psig
Post processing storage	3 years	0 – 60°C	no reversion	Ambient

The plan to develop a time/temperature indicator ink for MWS processing includes a sequence of:

1. Laboratory calibration to an irreversible color change of select co-topo-polymers after simulated (heated oil-bath) MWS-exposure for indicated duration at target processing temperatures.

2. Pilot-plant verification of the co-topo-polymer effect using pressure sensitive labels on packaged food pouches as they are microwave sterilized.

3. Commercial validation of the co-topo-polymer incorporated into a flexographic ink and reverse printed onto oriented polyester film to be laminated to the outside of functional barrier polymeric pouches by processing them in the MWS pilot-plant.

The initial two steps of the plan are addressed in this report.

Materials and Methods

Laboratory calibration

Proprietary formulations of the co-topo-polymeric pigments were prepared and milled into a high solids content "screen" ink vehicle. This ink was then applied with a hand proofer to a heat-resistant oriented polyester film coated with a pressure sensitive adhesive (PSA). The printed film was cut into approximately 4 cm long pieces and adhered to one end of thin aluminum strips, about 2 x 10 cm.

The printed film ends of the aluminum strips were dipped into a temperature-controlled (±1 °C) silicon oil bath for increasing intervals (10 seconds to 5 minutes, as measured by a stopwatch). These intervals were repeated at increasingly higher temperatures.

The strips were allowed to cool to ambient temperature and color-compared to each other and untreated strips. Two formulations were chosen for future evaluation (Table 2):

Table 2: TTI ink candidates

Property	Ink type A	Ink type B
Color Density	Very Dense	Less Dense
Initial Color	Dark Blue	Light Blue
Color change: 124°C; >4 min	magenta	pinkish magenta

Pilot-plant verification

Strips (approximately 3 x 7 cm) of the same PSA coated polyester film printed with the 2

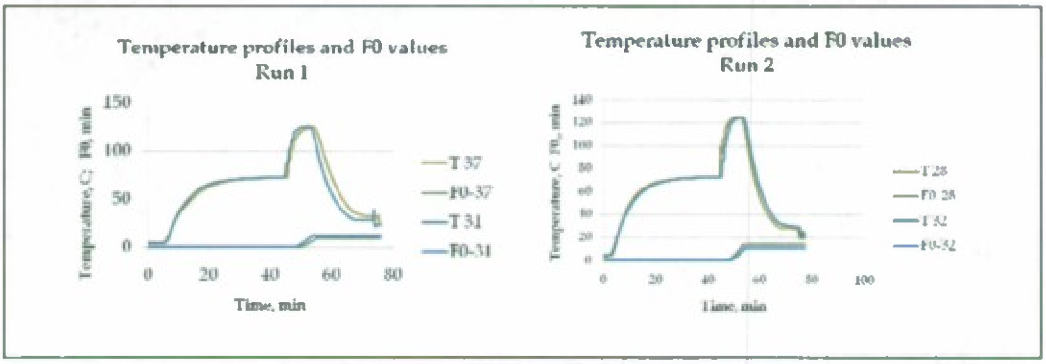

Figure A: MWS Process Conditions

best inks found in the calibration process were adhered to the outside of barrier all-polymeric pouches filled with salmon patties with Alfredo sauce. The pouches also contained the Ellab sensors used by WSU.

The MWS pilot plant is described in Tang et al. (2008). Specifically for this test, the MWS process was conducted using microwave power at 7.5, 7.5, 4.7 and 4.7 kW in the 4 cavities. Temperatures were set to: 72/124/123 °C for preheating, heating, and holding sections respectively with belt moving at 35 inch/min. Figure A summarizes the Time/temperature profile for the two runs with labeled pouches.

Results and Discussion

Laboratory calibration:

Color comparisons of the two preferred ink pigments are presented in Figure B. Each of the strips was submerged in the hot silicon oil bath for the time and temperature indicated. Each has two strips of printed film, one with ink A (left) and ink B (right). The image provided here presents the color of each strip after returning to room temperature. Here Ink A indicates little color change until about 50 seconds at 130°C. This change to magenta is clearly established by 120 seconds at 130°C, and too bright magenta by 180 seconds at 130°C. Ink B exhibited less reliable color dependability over the entire range of time and temperature, but demonstrated a clear change to pinkish magenta by 50 seconds exposure at 130°C. By 120 seconds at 130°C, the change was clear and noticeably differed from the untreated color.

These results were used to enhance the two formulations for the PSA labels sent to WSU for pilot plant runs there. (Actual formulation adjustments are proprietary, but are as indicated in the disclosures of Ribi (2010)).

Pilot-plant verification

Pre-run testing verified that no microwave field / ink interaction occurs.

Color comparisons of pouches labeled with the two ink pigments are presented in Figure C. Electronic thermocouples in the pouches were used to correlate the internal temperatures reached by the salmon with the external temperature of the pouches. Both inks can be optimized for distinct and irreversible color change following the selected time at the desired temperature.

Figure B: Color comparison of time temperature exposures of indicator inks

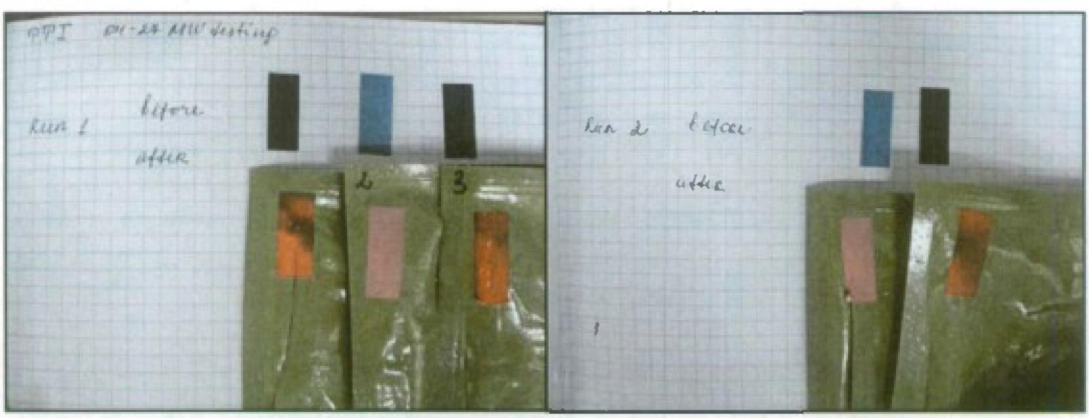

Figure C: Color comparison of TTI labels untreated and exposed to MWS conditions

Conclusions

Co-topo-polymeric compositions can be developed and adapted for the time and temperature process conditions present during microwave sterilization. A bench top procedure for screening and evaluation of compositions satisfactorily predicts behavior of the label in the MWS structure.

Advancing these findings to commercial printing processes requires:

- Incorporation of the co-topo-polymeric compositions into a heat resistant ink vehicle.
- Adjustment of the ink's viscosity to ink-metering requirements of the printing process.
- Compatibility of the reverse-printed dried ink film with the adhesive to be applied over it in a subsequent laminating step.

Research is currently underway to address these intermediate assessments and to begin production of printed, laminated high barrier all polymeric pouches.

References

Ratto, Jo Ann, J. Lucciarini, C. Thellen, D. Froio, and N. A. D'Souza, 2006, *The reduction of Solid Waste Associated with Military Ration Packaging*, , US Army Soldier System Center, Technical Report, Natick (Ma) TR-06/023. 75pp.

Ribi, H.O., 2010. Co-topo-polymeric compositions, Devices and Systems for Controlling Threshold and Delay Activation Sensitivities. US Patent App. # 2010/12018.

Tang Z., Mikhaylenko G., Liu F., Mah J.H., Tang J. , Pandit R., Younce F., 2008. *Microwave Sterilization of Sliced Beef in Gravy in 7-Oz Trays. Journal of Food Engineering, 89.* (4), 375-383.

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ANNEX D: nations

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ANNEX E:

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ANNEX F:

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Draft Quarterly Report

For the Period Ending
30 Sep, 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Quarterly Report

For the period ending
30 Sep 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Summary: After four quarters, the project is on budget but has experienced some slippage in important tasks. The deliverables 2 is ready to submit. Significant progress for deliverables 4 through 7 is reported here.

Project Overview

Objective

To advance the state of the art for Thermal Microwave Sterilization (MWS) for pouches of individual combat rations while maintaining current shelf life:

- improvements in the MWS process at Washington State University (WSU) and
- improvements in the non-foil light barrier materials considered in the Print-pack research

Deliverables

Task	Deliverable	Plan date	Act/Ant date
~~1~~	~~10 Laminations~~	~~31 Dec 2009~~	~~08 Jan 2010~~
2	Physical, Barrier, & Optical Data	28 Feb 2010	31 Oct 2010
~~3~~	~~Photodegradation Data~~	~~31 Jan 2010~~	~~5 Mar 2010~~
4	Retort & MWS Entrée Packages	30 Apr 2010	31 Mar 2011
~~5~~	~~Hot Fill Packages~~	~~30 Apr 2010~~	~~28 Sep 2010~~
6	Optimized MWS Entrée Packages	30 Jun 2010	31 May 2011
7	MWS Validation Report	30 Jun 2010	30 Jun 2011
8	Standard Condition Shelflife Modeling	30 Apr 2010	31 Oct 2010
9	Extreme Condition Shelflife Modeling	31 May 2010	30 Nov 2010
~~10~~	~~TTI Label Evaluation~~	~~30 Apr 2010~~	~~1 July 2010~~
11	Printed Pouch TTI Evaluation	31 Aug 2010	30 Nov 2010

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Accomplishments

1. Task 1 (Laminations): All laminations are completed; including advances include new barrier materials and alternate opacifying pigments for sealant films.
2. Task 2 (Material data sheets): Water vapor, oxygen, and light barrier and dielectric and physical properties have been determined for all of the task one laminations. (Data Sheets in ANNEX A.)
3. Task 3 (Photodegradation Data): The photodegradation assessment of the 10 sample laminations (GCMS/quantification of hexanal after extended light exposure indicated that several techniques for imparting light barrier functionality to pouches are feasible.
4. Tasks 4, 6, 7 (MWS process): Printpack and Washington State University (WSU) have negotiated a 9-month co-cost extension of their subcontract, and submitted this to DOD for approval. Details of proposed revision are provided in "Subcontracts" section. Submitting an FDA validation report for chicken and dumplings at the end of the second quarter of CY2011 is now planned. WSU has submitted its FDA validation report for a pouched Salmon & Alfredo sauce item.
5. Tasks 8 &9 (Shelf life Modeling): We will use the creator of the M-Rule food shelf model to calibrate the high barrier material characteristics in the model. This should allow completion of the shelf life scenarios over the next 60 days.
6. Tasks 10 & 11 (TTI Technology): Segan Industries (Burlingame, CA), subcontractor for the development of a flexographic ink providing Time Temperature Indications on microwave sterilized pouches has successfully evaluated its pigment in simulated laminations using in Printpack's temperature-resistant ink vehicle Planning for press trial and MWS evaluation are under way.

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Risks

Technical and program Risks

Technical Risks

1. Task 1 (Laminations): (None… task effectively complete)
2. Task 2 (None… task effectively complete):
3. Task 3 (Photodegradation Data) : (None… task effectively complete)
4. Tasks 4,6,7 (MWS process)

   Review of the previous US FDA validation of the WSU MWS process for mashed potatoes revealed the need to confirm that the packaging material used to contain the product did not adulterate it by leaching migrating chemicals in to food. *Risk mitigation:* Printpack has double-sided migration studies of the pouch materials for MWS in last year's chicken and dumplings and this year's Salmon & Alfredo sauce using the US FDA "Chemistry Guidelines" for its "Food Contact Material Notification" program (see ANNEX B).

   The WSU MWS processes for mashed potatoes and salmon & Alfredo defined acceptable methods for inoculating the product with appropriately thermal resistant- spores cold spot of the tray and pouched packages. Identifying the cold spot in a pouch of heterogeneous components is a similar process, each of the elements of the diverse chicken and dumpling food product has its distinct thermal heating properties. MWS process validation effort has identified the dielectric properties of its component foods. Next steps involved determining the package cold spot using a model food and developing the techniques for heat resistant spore inoculation. *Risk mitigation:* WSU personnel replaced two unreliable microwave generators on its pilot line and will begin to recalibrate the modified line to the original one in October 2010. They expect to complete cold spot determination in November. Monthly visits to the Pilot plant are planned to monitor activity and insure progress.

   Tasks 8,9 (Shelf life Modeling) The M-Rule Container Performance Model for Foods has been developed and validated foods and packaging materials on less complex than those involved in shelf stable combat rations. Its adoption to these systems requires an initial calibration of its calculations to the steady state conditions of the material's barrier performance (Transmission rates). *Risk mitigation:* Printpack will use the Model's developer to adapt materials characteristic inputs for these complex non-foil barrier laminations.

5. Tasks 10,11 (TTI Technology)

   TASK 10 (Immediate Solutions): (None… task effectively complete)

   TASK 11 (Intermediate Solutions)

   The time temperature indicator system used to print an indicator message on packaging material must resist not only premature development of in-

dicia during package material conversion, material shipment and storage, product packaging and sealing but also early message fade during post-processing shipment and storage. The formulation with its TTI functionality must be acceptable in Printpack's printing presses. *Risk mitigation:* Printpack and Segan complete the evaluation of three Segan chemistries in label form in WSU pilot plant runs. Segan prepared selected chemistries in Printpack's thermally-resistant ink vehicle and Segan developed a bench-top lamination simulation to anticipate their printing and laminating compatibility of this formulation must be acceptable in a commercial printing process:

Program Risks

1. Task 1 (Laminations): (None…this task is effectively complete)
2. Task 2 (Physical, Barrier, Optical Data): (None…this task is effectively complete)
3. Task 3 (Photodegradation Data): (None…this task is effectively complete)
4. Tasks 4,5,6,7 (Packaged Products): Printpack's packaging material is now available, but WSU faculty and staff have been focused on developing and submitting documentation for a salmon in Alfredo sauce product. Inoculated pouches of this product are now undergoing incubation. *WSU administration has committed to maintain the WSU MWS process pilot plant at its present location through the end of December, 2010, but they have requested a no-cost contract extension through the end of FY2011. The request to modify the DOD/Printpack contract is currently under review.*
5. Tasks 8 & 9 (Shelf life Modeling): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.
6. Tasks 10 &11 (TTI Technology):

   TASK 10 (Immediate Solutions): (None… task effectively complete)

   TASK 11 (Intermediate Solutions): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.

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Issues

Unexpected Issues

TASK AREA	COMMENT
1. Task 1 (Laminations):	(none)
2. Task 2 (Barrier data):	(none)
3. Task 3 (Photodegradation Data)	(none)
4. Tasks 4,6,7 (MWS process)	• The need for data confirming that the MWS process itself does not cause chemical changes in packaging materials that are otherwise compliant with US FDA requirements for high temperature sterilization was not recognized in the original project scope (or original FDA validation) Directed studies to provide such data (according to US FDA protocols*) have been obtained and are reported here as ANNEX B • A whole muscle item (salmon with Alfredo sauce has been added as a second FDA process validation subject. WSU has forwarded its Validation report to the US FDA.
5. Tasks 8,9 (Shelf life Modeling)	(none)
6. Tasks 10,11 (TTI Technology)	(none)

* http://www.fda.gov/Food/GuidanceComplianceRegulatoryInformation/GuidanceDocuments/FoodIngredientsandPackaging/ucm081818.htm. See especially "APPENDIX II. SELECTED MIGRATION TESTING PROTOCOLS"

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Details

Good News

TASK AREA	COMMENT
1. Task 1 (Laminations):	▪ *New improved WVTR films from Toppan (GL-ARHF) provided expected improved results.** ▪ OTR performance at or below NSRDEC targets from last year duplicated this year
2. Task 2 (Barrier data): 3. Task 3 (Photodegradation Data)	▪ *Photodegradation data indicates that 2 layers of pigmented adhesive is sufficient to protect lipids from photooxidation**
4. Tasks 4,6,7 (MWS process)	▪ *The WSU MWS pilot line will be available for trials throughout all of 2010.** ▪ *Polymeric laminations for food processed produced with improved visual quality and full functionality.** ▪ Pouches and roll stock for Salmon & Alfredo sauce successfully used for validation trials.
5. Task 10 (Immediate TTI Technology)	▪ *Work essentially complete and successful**
6. Tasks 11 (Intermediate TTI Technology)	▪ *Bench top testing of TTI pigment with standard flexographic heat-resistant vehicle confirmed functionality equal to or better than in previous screen printing vehicle** ▪ Bench top laminations successfully processed through WSU MWS process.

** Previously reported*

(ToC)

Details

Technical

TASK AREA	COMMENT
1. Task 1 (Laminations):	• Laminations complete (previously submitted).
2. Task 2 (Barrier data):	• Barrier data is summarized in ANNEX A.
3. Task 3 (Photodegradation Data)	• Photodegradation Data complete (previously submitted). • Other physical data is complete for the 10 laminations (submitted with ANNEX A). • WSU has completed its thin film MW resonance testing on the 10 laminations (previously submitted)
4. Tasks 4,6,7 (MWS process)	• The WSU MWS process has received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding. • The WSU MWS process has submitted a validation Report for Salmon & Alfredo using Printpack pouches. (The process previously received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding) • Printpack has successfully laminated all polymeric high barrier structures for both shelf-stable thermally-processed food and hot fill items. WSU is behind schedule with developing engineering data and inoculation methodology.
5. Tasks 8,9 (Shelf life Modeling)	• Barrier performance for laminations and raw material base films will be validated in model by model developer.
7. Task 10 (Immediate TTI Technology)	• Final report with successful results previously submitted.
8. Tasks 11 (Intermediate TTI Technology)	• In depth evaluations of precise and repeated color indications finished on bench top and onto the WSU Pilot Plant for additional confirmation. • Detailed preparations for press run are now in process at both Printpack and Segan.

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Details

Financial

Project Expenses as of 24 Sep 2010

COST ELEMENT	Contract Amt	Q-1 Amt	Q-2 Amt	Q-3 Amt	Q-4 Amt	Project to Date
Total Direct Labor	232,930	28,496	24,112	34,396	6,635	93,639
Payroll Tax & Benefits	84,511	10,339	8,748	12,497	2,389	33,973
Departmental Overhead	135,996	16,637	14,077	20,081	3,849	54,644
Labor Total	453,437	55,472	46,938	66,974	12,873	182,257
Consulting & Services	544,500	0	941	87,909	68,441	157,291
Materials & Plant Costs	70,850	33,170	-	16,153	158,940	208,263
Travel	24,530	6,660	4,152	11,705	1,980	24,497
Other Direct Costs	160,760	99,505	-6,807*	19,144	0	111,842
Total Costs	1,254,077	194,807	45,224	201,858	229,361	671,250
!0% Fee	125,409	19,481	4,522	20,186	22,936	67,125
Contract Total	1,379,486	214,288	49,746	222,044	252,297	738,375

** Adjustment for FY'08 Costs improperly charged to FY'09 Project.*

Project expenses to date as captured in Printpack's financial accounting system indicate that the work remains on budget. Some of the budgeted direct labor costs are reported here as plant costs. Much of the remaining work will be conducted by the subcontractors. Only one-third of the $400,000 plus WSU subcontract is reflected in the above costs.

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Equipment* ***(No change from last report)***

Equipment	Cost	Assoc. Task(s)	Location
OXTRAN® 2/21 SL Env. Chamber Operating. System	$61,641	2	Printpack Analytical Services Lab 5 Barber Industrial Ct. Villa Rica, GA 30180
Leap Autosampler System	$36,107.	3	

* No additional equipment purchases during 2nd, 3rd, or 4th quarters

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SUBCONTRACTS

Subcontracts

Subcontractor	Cost	Assoc. Task(s)	Status
Washington State University	$400,533	4,6,7	Work underway; no-cost time extension negotiated with WSU and proposed to DOD
Segan Industries	$144,000.	10,11	Phase 1 complete; work on Phase II underway; on budget. Timing delayed by availability of WSU pilot plant

Amended timing for WSU deliverables as negotiated with Printpack and proposed to DOD is…

Deliverables	Delivery Date
1. Report on thermal stability of selected TTI label materials in high temperature environment suitable for thermal sterilization applications. We may use WSU package film test cells and oil baths to conduct heating tests. Samples will be evaluated at WSU and in Printpack. (See ANNEX C)	Oct 31,2010
2. Report on interaction among microwaves, food package films and foods; with evaluation of thermal stability of Printpack films using 40 kW 915 MHz microwave sterilization system at WSU. Send processed films to Printpack for quality evaluation. Formulation of the standard test food model will be developed in cooperation with Printpack. (See ANNEX D)	Oct 31,2010
3. Developed and validated thermal processing procedures for two food products (chicken dumpling and salmon in Alfredo sauce) in three selected film materials using the 915 MHz microwave sterilization system.	June 30,2011
4. Produce food products in pouches, conduct microbial check, and send 40 pouches of each product to Natick for sensory and shelf-life studies. We will also process the same foods in MRE foil pouches using conventional retorting method for comparison.	Mar 31,2011
5. Assistance to Printpack in studies of commercial scale-up abilities.	Jul 31,2011
6. Assistance to Printpack in testing TTI system for package materials compatible with microwave sterilization system.	Nov 30,2010

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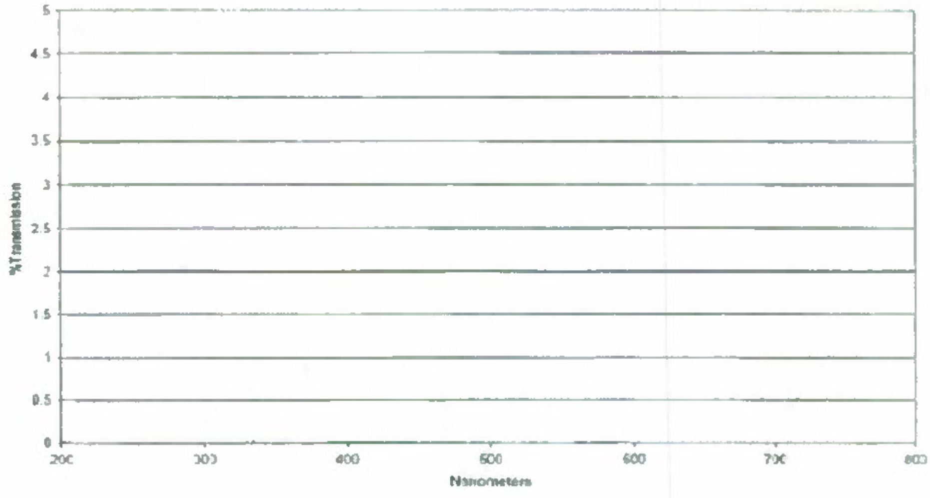
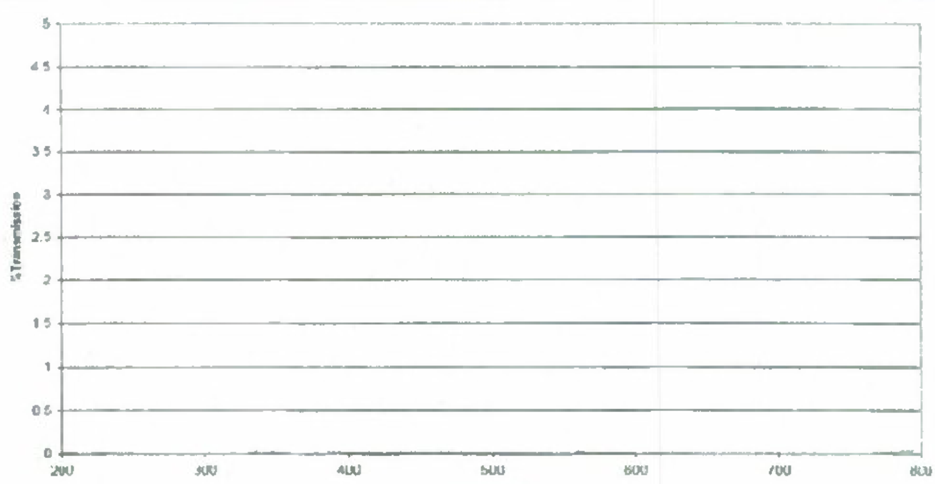
Data Sheets:

Properties:
Physical
Barrier
And
Optical

STRUCTURE:	OPET/Kur-C/Kur-N/Cblack B343-997			
		Structure No. 1		
PROPERTY		UNITS	METHOD	VALUE
Gauge		micron	ASTM F2251	120
Yield		m^2 / Kg	ASTM D4321	7.56
Basis Weight		gm / m^2	ASTM D646	132.248
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	100
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	23,870
	CMD			25,197
Elongation @ break	MD	%	ASTM D882	137
	CMD			120
1% Secant Modulus	MD	N / mm^2	ASTM D882	1627.86523
	CMD			1413.39112
Elmendorf Tear (notched)	MD	gm	ASTM D689	172
	CMD			172
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.214
	in/in			0.117
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	120.0787
Heat Seal Strength	250 F	gm / 25 mm	ASTM F88	11,291
WVTR-37.8°C-90% RH	flat	gm·day/m^2	ASTM F1249	4.061
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	3.762
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	4.233
OTR-23°C-90% RH	flat	cc·day/m^2	ASTM D3985	0.009
OTR-23°C-90% RH	5 gelbo		ASTM D3985	0.332
OTR-23°C-90% RH	10 gelbo		ASTM F392	0.567
OTR-23°C-0% RH	flat	cc·day/m^2	ASTM D3985	0.009
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.023
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.040



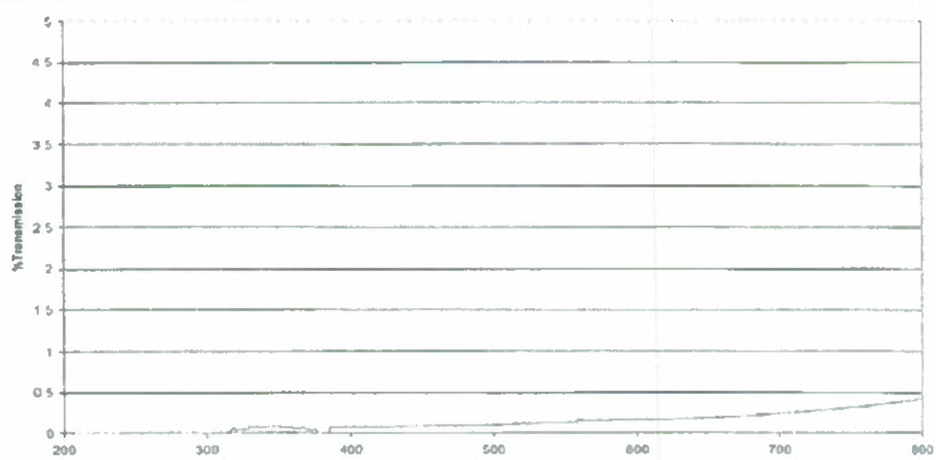
STRUCTURE:	OPET/Kur-C/Kur-N/CMYW B343T-997			
Structure No. 2				
PROPERTY		**UNITS**	**METHOD**	**VALUE**
Gauge		micron	ASTM F2251	119
Yield		m^2 / Kg	ASTM D4321	7.17
Basis Weight		gm / m^2	ASTM D646	139.5
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	100
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	25,281
	CMD			24,865
Elongation @ break	MD	%	ASTM D882	139
	CMD			104
1% Secant Modulus	MD	N / mm^2	ASTM D882	1742.6624
	CMD			1684.30556
Elmendorf Tear (notched)	MD	gm	ASTM D689	198
	CMD			252
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.24
	in/in			0.348
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	146.6535
Heat Seal Strength	260 F	gm / 25 mm	ASTM F88	2,371
WVTR-37.8°C-90% RH	flat	gm·day/m^2	ASTM F1249	4.244
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	3.963
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	4.165
OTR-23°C-90% RH	flat	cc·day/m^2	ASTM D3985	0.009
OTR-23°C-90% RH	5 gelbo		ASTM D3985	0.184
OTR-23°C-90% RH	10 gelbo		ASTM F392	0.192
OTR-23°C-0% RH	flat	cc·day/m^2	ASTM D3985	0.009
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.023
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.045



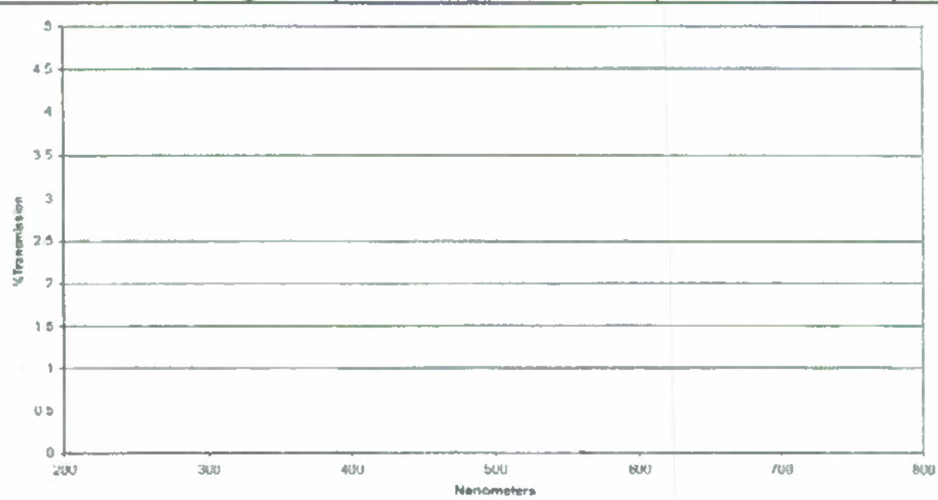
STRUCTURE: OPET/Kur-C/Kur-N/Clear B343-997

Structure No. 3

PROPERTY		UNITS	METHOD	VALUE
Gauge		micron	ASTM F2251	129
Yield		m² / Kg	ASTM D4321	7.30
Basis Weight		gm / m²	ASTM D646	137.0
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	86.9
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	26,045
	CMD			25,231
Elongation @ break	MD	%	ASTM D882	114.0
	CMD			25560.0
1% Secant Modulus	MD	N / mm²	ASTM D882	1558.4
	CMD			119.1
Elmendorf Tear (notched)	MD	gm	ASTM D689	246
	CMD			236
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.47
	in/in			0.45
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	623
Heat Seal Strength	330 F	gm / 25 mm	ASTM F88	6,542
WVTR-37.8°C-90% RH	flat	gm·day/m²	ASTM F1249	4.405
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	4.255
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	3.514
OTR-23°C-90% RH	flat	cc·day/m²	ASTM D3985	0.009
OTR-23°C-90% RH	5 gelbo		ASTM D3985	0.398
OTR-23°C-90% RH	10 gelbo		ASTM F392	0.295
OTR-23°C-0% RH	flat	cc·day/m²	ASTM D3985	0.009
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.045
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.042



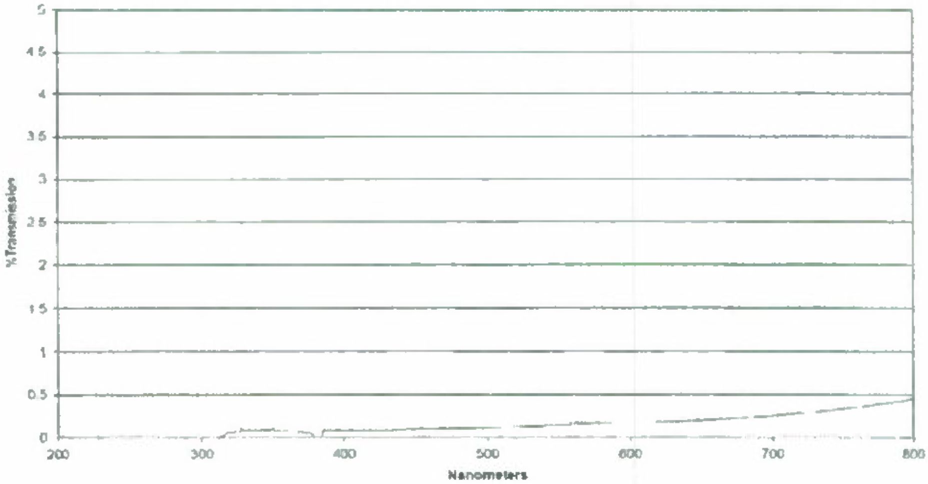
STRUCTURE:	OPET/Kur-C/Kur-N/C Black B343T-997			
	Structure No. 4			
PROPERTY		**UNITS**	**METHOD**	**VALUE**
Gauge		micron	ASTM F2251	125
Yield		m² / Kg	ASTM D4321	7.25
Basis Weight		gm / m²	ASTM D646	137.9
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	100.0
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	25,265
	CMD			24,973
Elongation @ break	MD	%	ASTM D882	136.0
	CMD			145.0
1% Secant Modulus	MD	N / mm²	ASTM D882	1450.1
	CMD			1406.4
Elmendorf Tear (notched)	MD	gm	ASTM D689	201
	CMD			243
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.30
	in/in			0.14
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	111
Heat Seal Strength	280 F	gm / 25 mm	ASTM F88	4,888
WVTR-37.8°C-90% RH	flat	gm·day/m²	ASTM F1249	3.767
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	3.889
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	3.007
OTR-23°C-90% RH	flat	cc·day/m²	ASTM D3985	0.068
OTR-23°C-90% RH	5 gelbo		ASTM D3985	4.104
OTR-23°C-90% RH	10 gelbo		ASTM F392	4.002
OTR-23°C-0% RH	flat	cc·day/m²	ASTM D3985	0.431
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.386
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.372



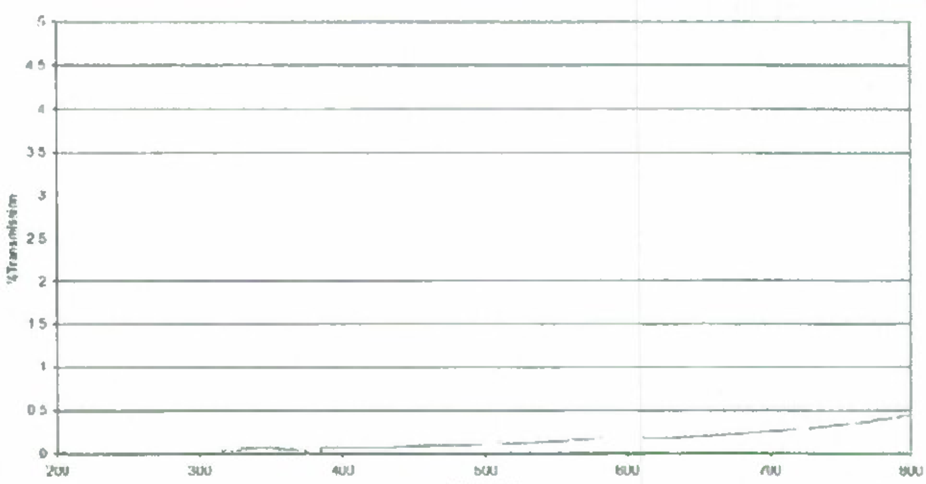
STRUCTURE:	OPET/GL-PET-ARH/Kur-N/Clear B343-997			

Structure No. 5

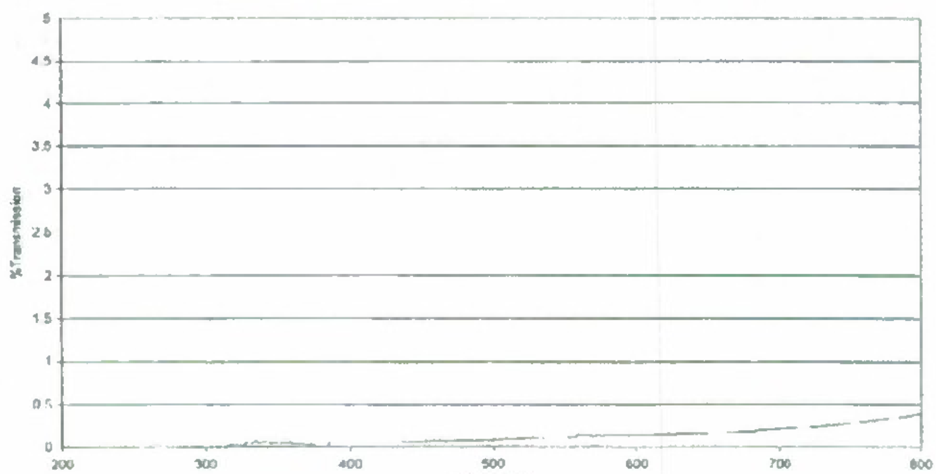
PROPERTY		UNITS	METHOD	VALUE
Gauge		micron	ASTM F2251	131
Yield		m² / Kg	ASTM D4321	7.32
Basis Weight		gm / m²	ASTM D646	136.7
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	85.5
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	21,433
	CMD			25,394
Elongation @ break	MD	%	ASTM D882	133.0
	CMD			115.0
1% Secant Modulus	MD	N / mm²	ASTM D882	1680.4
	CMD			1464.1
Elmendorf Tear (notched)	MD	gm	ASTM D689	262
	CMD			256
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.47
	in/in			0.46
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	713
Heat Seal Strength	320 F	gm / 25 mm	ASTM F88	3,765
WVTR-37.8°C-90% RH	flat	gm day/m²	ASTM F1249	0.344
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	0.580
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	0.595
OTR-23°C-90% RH	flat	cc day/m²	ASTM D3985	0.009
OTR-23°C-90% RH	5 gelbo		ASTM D3985	0.316
OTR-23°C-90% RH	10 gelbo		ASTM F392	0.198
OTR-23°C-0% RH	flat	cc day/m²	ASTM D3985	0.009
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.053
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.031



STRUCTURE:	OPET/GL-PET-ARHF/Kur-N/Clear B343-997			
		Structure No. 6		
PROPERTY		UNITS	METHOD	VALUE
Gauge		micron	ASTM F2251	128
Yield		m² / Kg	ASTM D4321	7.33
Basis Weight		gm / m²	ASTM D646	136.5
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	87.8
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	25,708
	CMD			24,595
Elongation @ break	MD	%	ASTM D882	157.0
	CMD			103.0
1% Secant Modulus	MD	N / mm²	ASTM D882	1564.4
	CMD			1582.4
Elmendorf Tear (notched)	MD	gm	ASTM D689	214
	CMD			230
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.45
	in/in			0.47
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	702
Heat Seal Strength	330 F	gm / 25 mm	ASTM F88	3,338
WVTR-37.8°C-90% RH	flat	gm day/m^2	ASTM F1249	0.158
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	0.555
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	0.490
OTR-23°C-90% RH	flat	cc day/m^2	ASTM D3985	0.009
OTR-23°C-90% RH	5 gelbo		ASTM D3985	0.118
OTR-23°C-90% RH	10 gelbo		ASTM F392	0.253
OTR-23°C-0% RH	flat	cc day/m^2	ASTM D3985	0.009
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.020
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.079



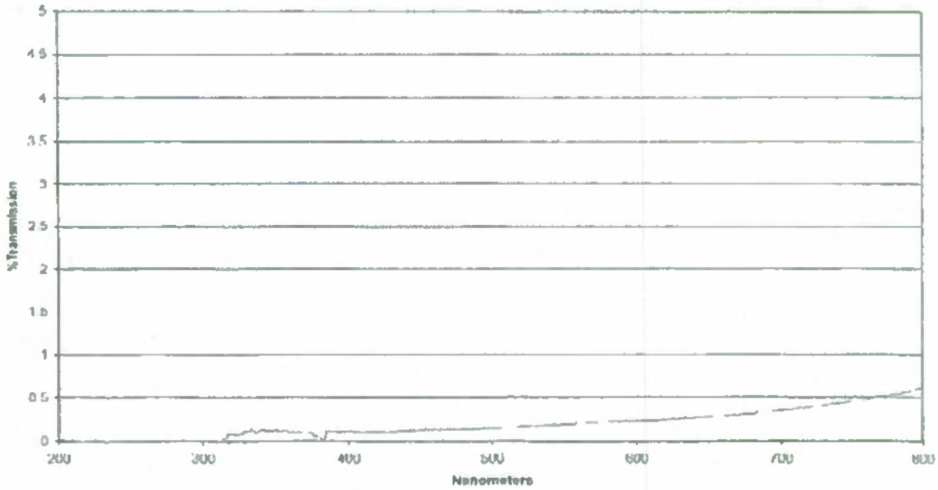
STRUCTURE:	OPET/WSX 03 Y07/Kur-N/Clear B343-997			
Structure No. 7				
PROPERTY		**UNITS**	**METHOD**	**VALUE**
Gauge		micron	ASTM F2251	129
Yield		m^2 / Kg	ASTM D4321	7.32
Basis Weight		gm / m^2	ASTM D646	136.6
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	88.7
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	24,540
	CMD			28,108
Elongation @ break	MD	%	ASTM D882	115.0
	CMD			108.0
1% Secant Modulus	MD	N / mm^2	ASTM D882	1666.5
	CMD			1543.9
Elmendorf Tear (notched)	MD	gm	ASTM D689	230
	CMD			275
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.40
	in/in			0.44
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	561
Heat Seal Strength	320 F	gm / 25 mm	ASTM F88	4,123
WVTR-37.8°C-90% RH	flat	gm·day/m^2	ASTM F1249	3.185
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	3.201
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	1.176
OTR-23°C-90% RH	flat	cc·day/m^2	ASTM D3985	0.009
OTR-23°C-90% RH	5 gelbo		ASTM D3985	0.056
OTR-23°C-90% RH	10 gelbo		ASTM F392	3.212
OTR-23°C-0% RH	flat	cc·day/m^2	ASTM D3985	0.014
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.009
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.271



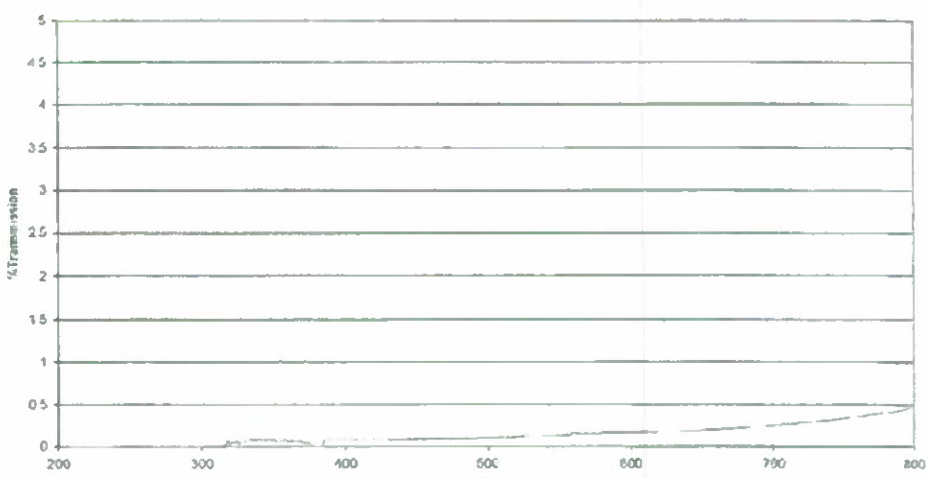
STRUCTURE:	OPET/GL-PET-ARHF/BON/Clear B343-997		

Structure No. 8

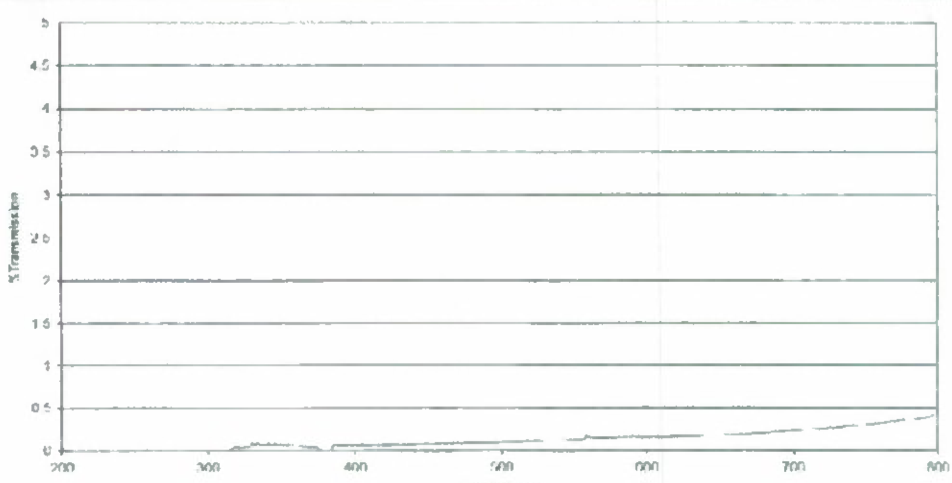
PROPERTY		UNITS	METHOD	VALUE
Gauge		micron	ASTM F2251	129
Yield		m² / Kg	ASTM D4321	7.45
Basis Weight		gm / m²	ASTM D646	134.2
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	81.4
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	26,950
	CMD			24,579
Elongation @ break	MD	%	ASTM D882	133.0
	CMD			123.0
1% Secant Modulus	MD	N / mm²	ASTM D882	1594.6
	CMD			1530.2
Elmendorf Tear (notched)	MD	gm	ASTM D689	230
	CMD			217
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.44
	in/in			0.47
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	585
Heat Seal Strength	320 F	g / 25 mm	ASTM F88	4,764
WVTR-37.8°C-90% RH	flat	gm·day/m²	ASTM F1249	0.229
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	4.557
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	0.420
OTR-23°C-90% RH	flat	cc·day/m²	ASTM D3985	0.147
OTR-23°C-90% RH	5 gelbo		ASTM D3985	1.555
OTR-23°C-90% RH	10 gelbo		ASTM F392	1.293
OTR-23°C-0% RH	flat	cc·day/m²	ASTM D3985	0.180
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.660
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.516



STRUCTURE:	OPET/Kur-C/BON/Clear B343-997			
Structure No. 9				
PROPERTY		**UNITS**	**METHOD**	**VALUE**
Gauge		micron	ASTM F2251	128
Yield		m² / Kg	ASTM D4321	7.38
Basis Weight		gm / m²	ASTM D646	135.5
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	85.3
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	28,995
	CMD			24,277
Elongation @ break	MD	%	ASTM D882	140.0
	CMD			146.0
1% Secant Modulus	MD	N / mm²	ASTM D882	1530.1
	CMD			1432.4
Elmendorf Tear (notched)	MD	gm	ASTM D689	198
	CMD			198
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.43
	in/in			0.46
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	462
Heat Seal Strength	320 F	gm / 25 mm	ASTM F88	11,010
WVTR-37.8°C-90% RH	flat	gm·day/m²	ASTM F1249	4.464
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	4.249
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	4.214
OTR-23°C-90% RH	flat	cc·day/m²	ASTM D3985	0.023
OTR-23°C-90% RH	5 gelbo		ASTM D3985	1.719
OTR-23°C-90% RH	10 gelbo		ASTM F392	1.677
OTR-23°C-0% RH	flat	cc·day/m²	ASTM D3985	0.237
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.175
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.073



STRUCTURE:	OPET/Kur-C/Clear B343-997			
		Structure No. 10		
PROPERTY		**UNITS**	**METHOD**	**VALUE**
Gauge		micron	ASTM F2251	111
Yield		m² / Kg	ASTM D4321	8.87
Basis Weight		gm / m²	ASTM D646	112.7
Gloss @ 45°		%	ASTM D2457	
Haze		%	ASTM D1003	n/a
Opacity		%	ASTM D589	88.9
Tensile Strength @ break	MD	kg / 25 mm	ASTM D882	4,992
	CMD			5,219
Elongation @ break	MD	%	ASTM D882	606.0
	CMD			702.0
1% Secant Modulus	MD	N / mm²	ASTM D882	1487.1
	CMD			1412.3
Elmendorf Tear (notched)	MD	gm	ASTM D689	234
	CMD			246
Coefficient of Friction (kinetic)	out/out	gm vertical/gm lateral	ASTM D1894	0.42
	in/in			0.46
Hot Tack Strength	300 F	gm / 25 mm	ASTM F1921	1210
Heat Seal Strength	320 F	gm / 25 mm	ASTM F88	3,545
WVTR-37.8°C-90% RH	flat	gm·day/m²	ASTM F1249	4.596
WVTR-37.8°C-90% RH	5 gelbo		ASTM F1249	4.504
WVTR-37.8°C-90% RH	10 gelbo		ASTM F392	4.368
OTR-23°C-90% RH	flat	cc·day/m²	ASTM D3985	0.009
OTR-23°C-90% RH	5 gelbo		ASTM D3985	0.358
OTR-23°C-90% RH	10 gelbo		ASTM F392	0.732
OTR-23°C-0% RH	flat	cc·day/m²	ASTM D3985	0.045
OTR-23°C-0% RH	5 gelbo		ASTM D3985	0.034
OTR-23°C-0% RH	10 gelbo		ASTM F392	0.009



Qualitative Migration Data:

Control (unused Pouch)
Pouched retorted with WPG & Alfredo Sauce
Pouched MW sterilized with WPG & Alfredo Sauce

```
File        :C:\msdchem\1\DATA\8235684\100908\02.D
Operator    :
Acquired    :  8 Sep 2010  15:42     using AcqMethod 8235684A.M
Instrument  :   FDA GC 41
Sample Name: 6-2798-1 Headspace 4in²
Misc Info   :
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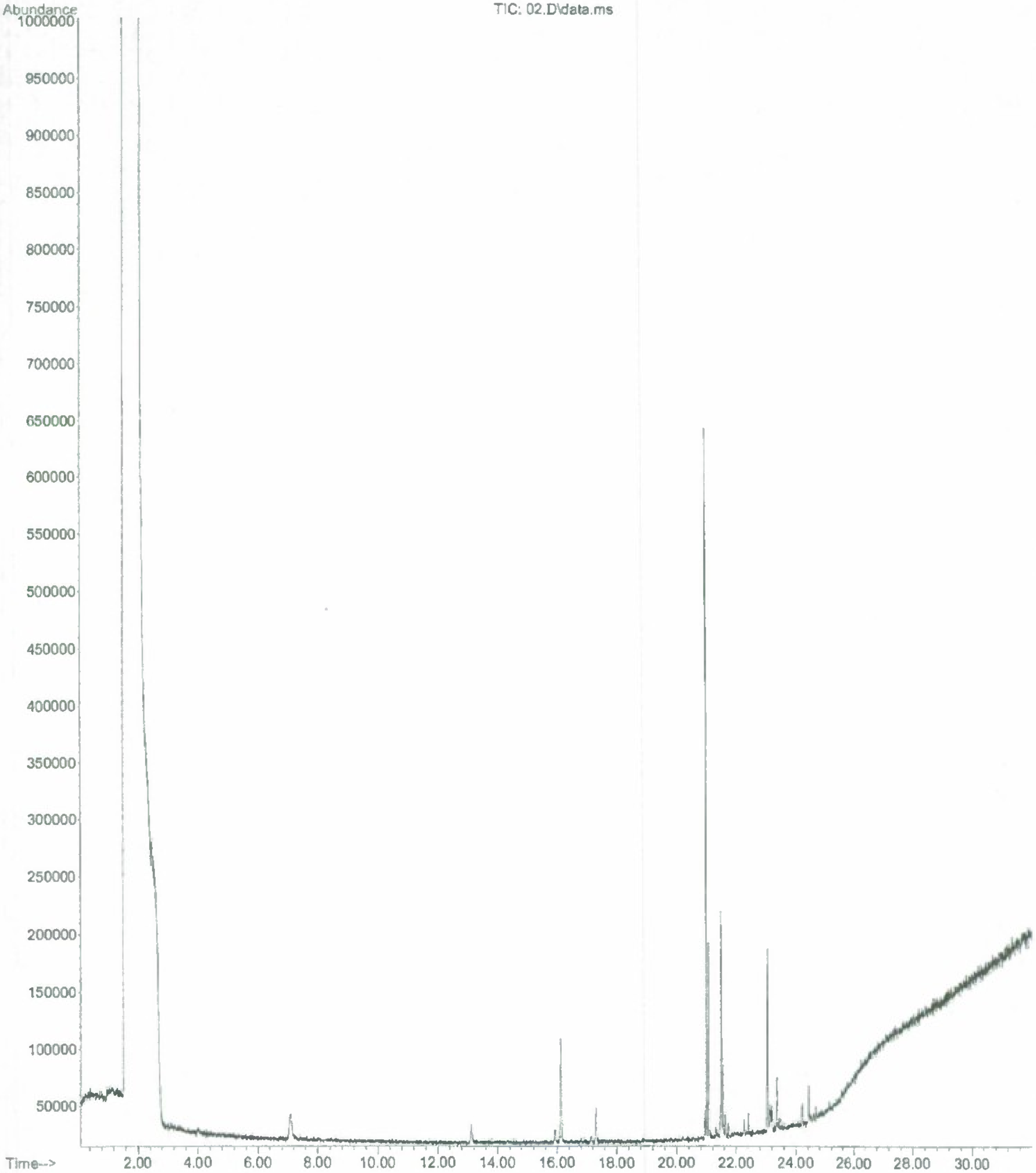

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Sample Name: 6-2799-2 Headspace 4in²
Misc Info   :
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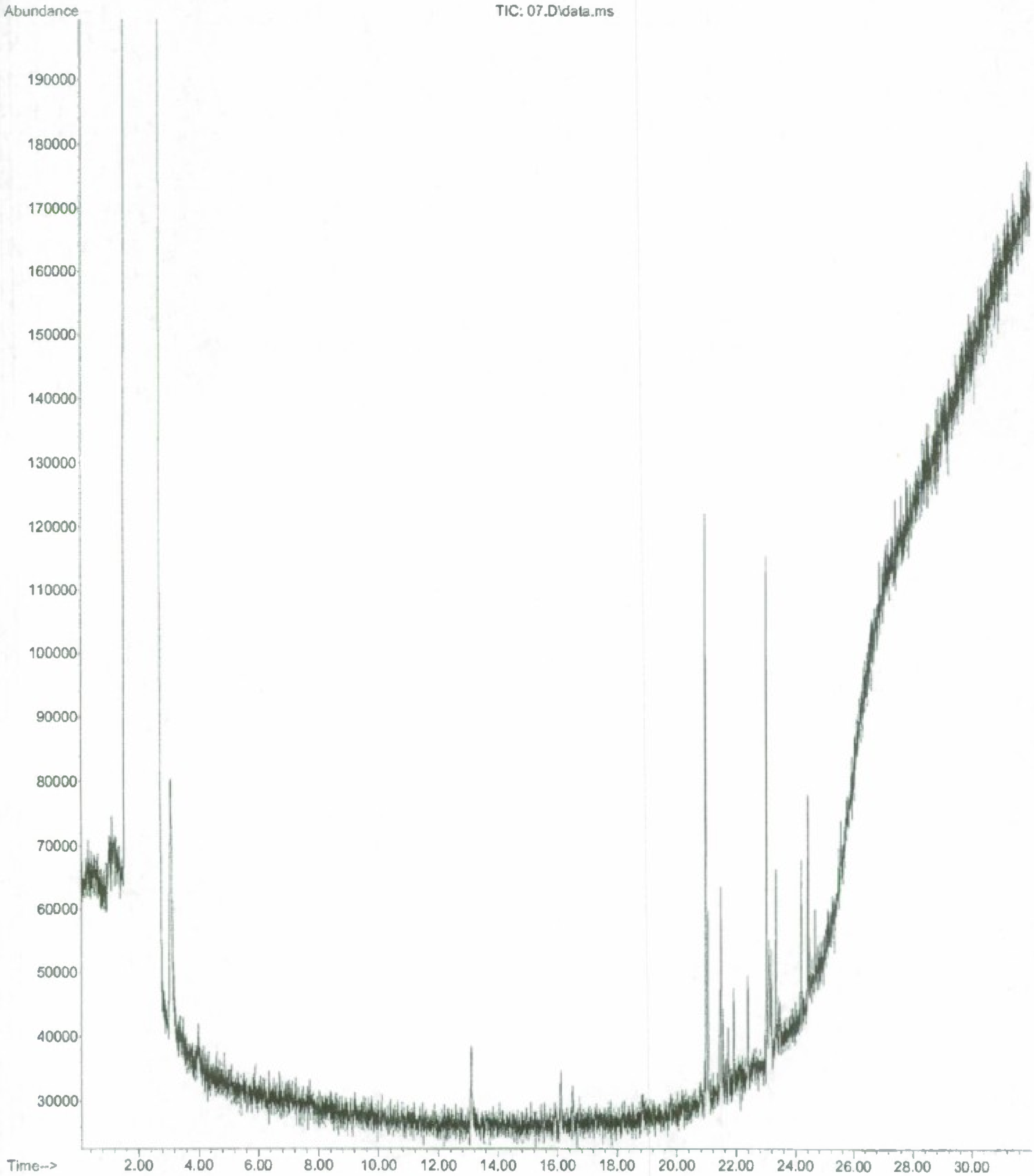

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Instrument  :    FDA GC 41
Sample Name: 6-2800-2 Headspace 4in²
Misc Info   :
Vial Number: 11
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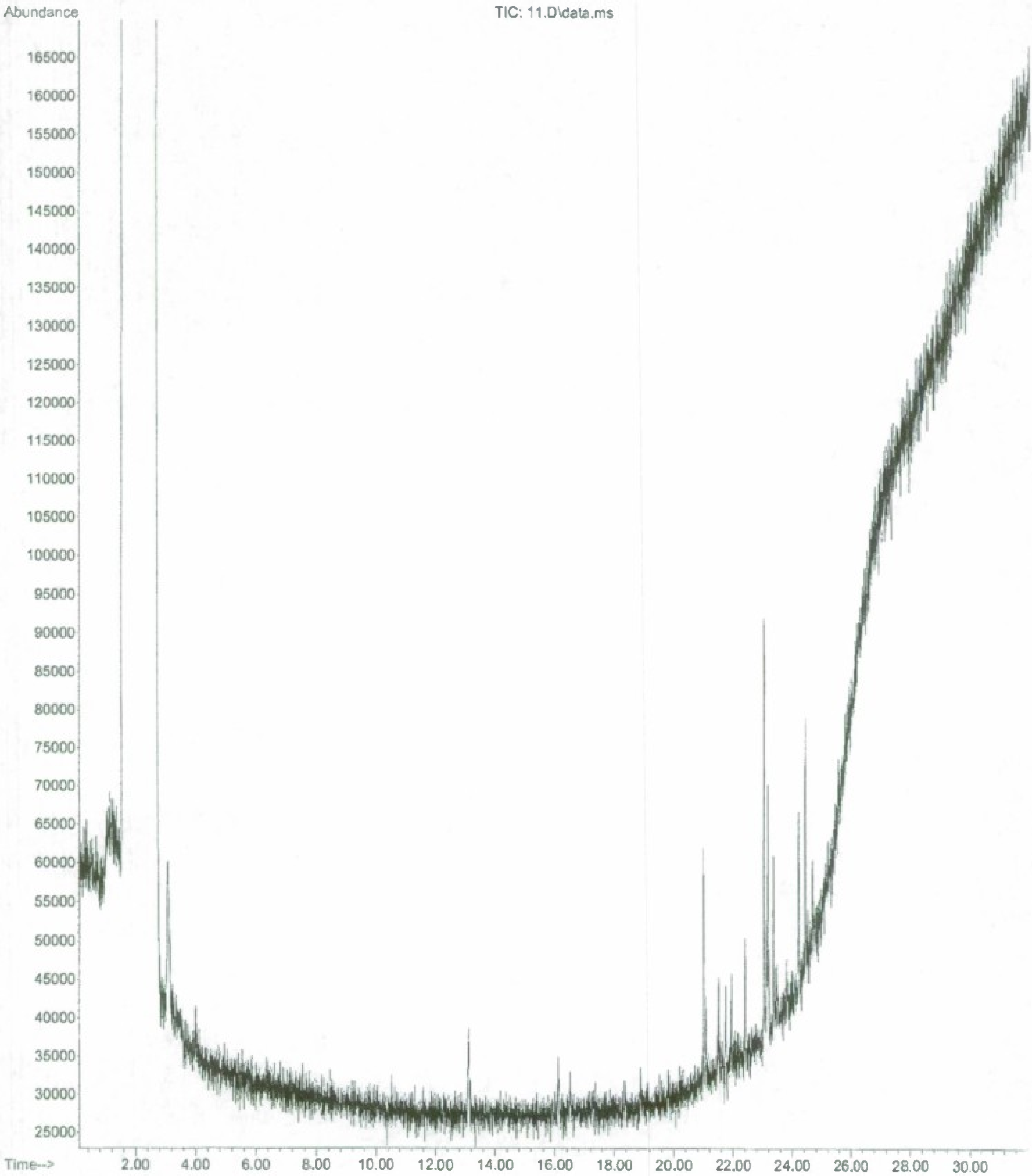


TTI label materials

In high temperature environment
Suitable for thermal sterilization applications

TTI testing in MW system

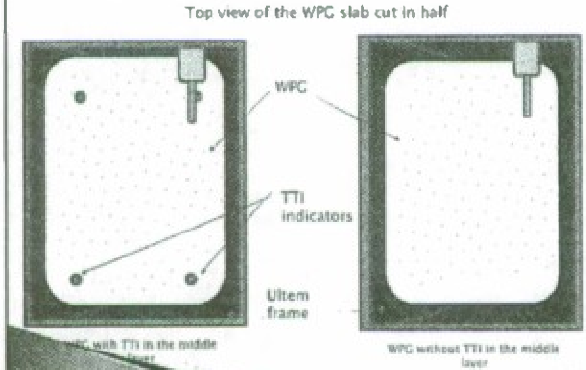
Printpack project
Aug 2010

Experiment to address concerns that TTI may interfere with heating pattern or Ellab readings

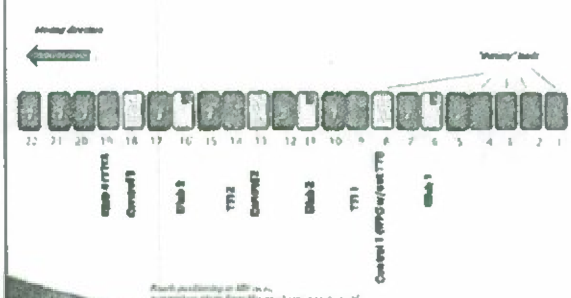
- Objective: study heating pattern of the whey protein gel (WPG) with and without presence of time-temperature indicators (TTI)
- Experiment:
  - Process WPG (Recipe GM 27) with and without TTI in MW without holding
  - Place the TTI in the middle layer of the WPG (as depicted on Slide 3)
  - Loading setup: process WPG w/out and with TTI in the middle layer (as depicted on Slide 4), in at least duplicate
  - Evaluate heating pattern of WPGs with and w/out TTI

Proposed plan for TTI test Aug 2010

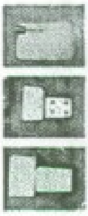
Top view of the WPG slab cut in half



Positioning of the pouches in the MW system



Experiment nomenclature and setup



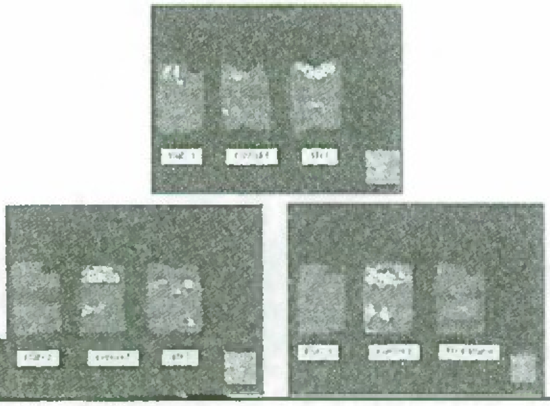
- Ellab: pouch contains Ellab sensor put in the middle of two WPG layers
- TTI: pouch contains 4 types of TTI put in the middle of two WPG layers
- Control: pouch contains WPG cut in the middle, no sensors or TTI used

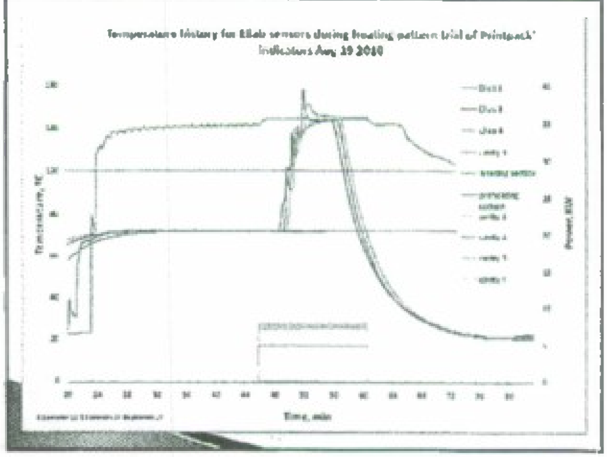
- All experimental pouches and dummy loads were approximately of the same size and weight

MW system setup

- 8-oz PrintPak® pouch
- MW power set: 8.1 / 8.2 / 4.7 / 4.7 kW for 4 MW heating cavities
- Actual MW power delivered: 7.6 / 7.3 / 4.7 / 4.7 kW for 4 MW heating cavities
- Pre-heating time: 30 min
- Speed: 42 inch/min (MW heating time: 3 min)
- Water temperature: 72 / 124 / 123°C for preheating, MW heating and holding sections
- System pressure: 26 psig
- Cooling time: 5 min

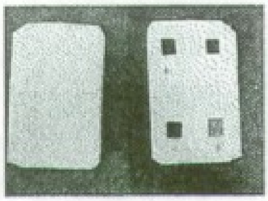
Heating patterns for WPGs with Ellab sensors, TTI and controls (no sensors)





Observations

- During heating in MW systems the WPG that had a rubber band holding two layers together got deformed (perhaps due to higher rubber bend shrinkage then that of WPG).



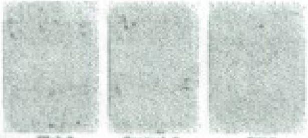
Position of rubber band

Before testing ***After MW testing***

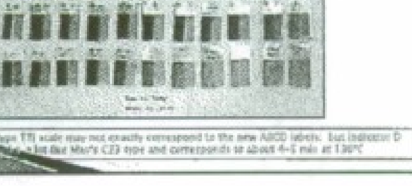
- Possible solution: do not use rubber band to hold layers together, instead try a paper tape

Observations

- Analysis of heating pattern did not reveal major changes in location of cold spots among WPCs with or without the TTI (Slide 7)
- It was noted that heating patterns for Ellab 2, 3, & 4 were slightly different in intensity compared to control and TTI in neighboring positions:
  - The heating pattern for Ellab 2, 3 and 4 was similar for control or TTI samples (Slide 7), however, intensity of the color development was a little less suggesting possibly lower temperature-time exposure to high temperatures
  - The Ellab 1 did not show noticeable deviation in time-temperature history as judged by color development for TTI1 and Control 1. It was also noted that Ellab 1 stopped working during MW run, temperature history for this Ellab was lost.



Ellab 2 Control 2 TTI 2



TTI from Aug 2010 MW testing, max T°C for D3 was 131.2°C

Old scale for May 2010 TTI

...old type TTI scale may not exactly correspond to the new ABCD labels. But indicator D ... a bit like Mar's C23 type and corresponds to about 4-5 min at 130°C

Conclusions

- Presence of TTI inside the packaged food does not influence the heating pattern of WPG or intensity of the cold spot heating
  - It was observed that 3 out of 4 Ellabs produced lower intensity heating pattern compared to neighboring WPG samples. The computer vision method is very sensitive. One has to run many samples in order to provide information about the range of RGB value that would indicate that one sample had significantly lower heating compared to the other. The observed lower intensity of heating pattern may not translate into a significant difference in time-temperature treatment unless sufficient amount of observations is collected
- Presence of TTI next to the sensor tip of the Ellab sensor did not influenced the heating pattern or altered the Ellab sensor readings
- It is safe to include Printpack TTI into further testing of materials that would undergo MW treatment

ANNEX D

Thermal Stability of Printpack Films
Using WSU 40 kW 915 MHz Microwave Sterilization System

PRINTPACK DOD FILMS THERMAL STABILITY IN HIGH TEMPERATURE ENVIRONMENT

Printpack project
Proposal task 3
April - May 2010

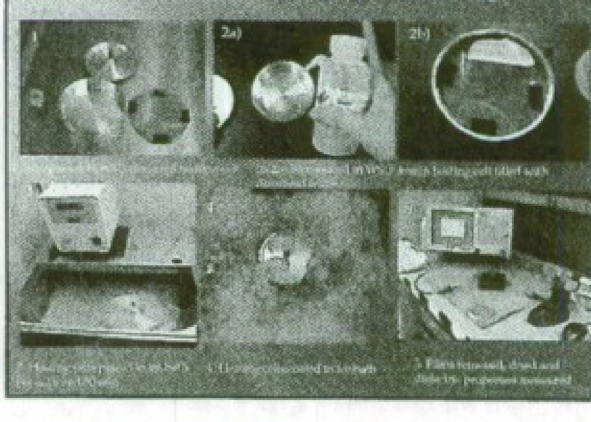
Scope

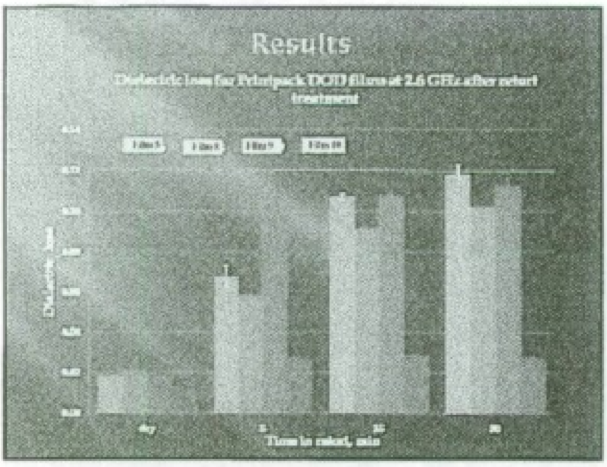
- Thermal stability of selected by Printpack films was assessed by changes in dielectric properties of films after treatment in the environment simulating retort conditions

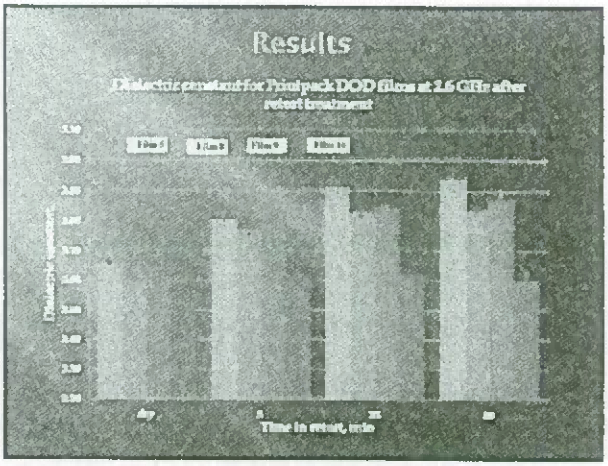
Materials and methods

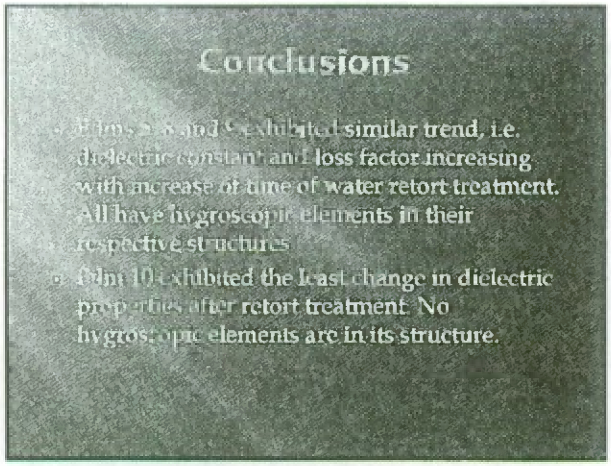
- Four DOD films (#5, 8, 9 and 10) were tested in the oil bath set at 121.1 °C for 5, 15 and 30 min
- Dielectric properties of these films were evaluated in at least 2 replicates for each time increment. Two data points were collected for each one of the replicates

Materials and methods




Results
Dielectric loss for Printpack DOD films at 2.6 GHz after retort treatment
Time in retort, min


Results
Dielectric constant for Printpack DOD films at 2.6 GHz after retort treatment
Time in retort, min


Conclusions
Films 5, 8 and 9 exhibited similar trend, i.e. dielectric constant and loss factor increasing with increase of time of water retort treatment. All have hygroscopic elements in their respective structures
Film 10 exhibited the least change in dielectric properties after retort treatment. No hygroscopic elements are in its structure.

Draft Quarterly Report

For the Period Ending
31 Dec, 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Quarterly Report

For the period ending
31 Dec 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Summary: After five quarters, delays at the major subcontractor, Washington State University, have precluded progress for several tasks in this project. Major progress has been made in preparing predictive shelf life models. (Standard and Extreme logistics conditions)

Contents

Project Overview

Objective
To advance the state of the art for Thermal Microwave Sterilization (MWS) for pouches of individual combat rations while maintaining current shelf life:

- improvements in the MWS process at Washington State University (WSU) and
- improvements in the non-foil light barrier materials considered in the Printpack research

Deliverables

Task	Deliverable	Plan date	Act/Ant date
~~1~~	~~10 Laminations~~	~~31 Dec 2009~~	~~08 Jan 2010~~
~~2~~	~~Physical, Barrier, & Optical Data~~	~~28 Feb 2010~~	~~31 Oct 2010~~
~~3~~	~~Photodegradation Data~~	~~31 Jan 2010~~	~~5 Mar 2010~~
4	Retort & MWS Entrée Packages	30 Apr 2010	31 Mar 2011
~~5~~	~~Hot Fill Packages~~	~~30 Apr 2010~~	~~28 Sep 2010~~
6	Optimized MWS Entrée Packages	30 Jun 2010	31 May 2011
7	MWS Validation Report	30 Jun 2010	30 Jun 2011
8	Standard Condition Shelflife Modeling	30 Apr 2010	31 Dec 2010
9	Extreme Condition Shelflife Modeling	31 May 2010	31 Dec 2010
~~10~~	~~TTI Label Evaluation~~	~~30 Apr 2010~~	~~1 July 2010~~
11	Printed Pouch TTI Evaluation	31 Aug 2010	31 Mar 2011

Accomplishments

1. Task 1 (Laminations): All laminations are completed; including advances include new barrier materials and alternate opacifying pigments for sealant films.
2. Task 2 (Material data sheets): Water vapor, oxygen, and light barrier and dielectric and physical properties have been determined for all task 1 laminations.
3. Task 3 (Photodegradation Data): The photodegradation assessment of the 10 sample laminations (GCMS/quantification of hexanal after extended light exposure indicated that several techniques for imparting light barrier functionality to pouches are feasible.)
4. Tasks 4, 6, 7 (MWS process): Printpack and Washington State University (WSU) have negotiated a 9-month co-cost extension of their subcontract, and submitted this to DOD for approval. Details of proposed revision are provided in "Subcontracts" section. Submitting an FDA validation report for chicken and dumplings at the end of the second quarter of CY2011 is now planned. WSU submitted its FDA validation report for a pouched Salmon & Alfredo sauce item .
5. *Tasks 8 &9 (Shelf life Modeling)*:* Assistance from the author of the M-Rule food shelf model allowed calibration of the barrier transmission rates of the base films and composite lamination. Data input for chicken and dumplings entrée is now finished and initial programming for the standard shelf life scenario completed. Deliverables for these to tasks planned for year's end.
6. Tasks 10 & 11 (TTI Technology): Press trial and lamination are planned for December. MWS evaluation depends on WSU schedule.

** Immediate past quarter's accomplishment*

Risks

Technical and program Risks

Technical Risks

1. Task 1 (Laminations): (None… task complete)
2. Task 2 (None… task complete):
3. Task 3 (Photodegradation Data) : (None… task complete)
4. Tasks 4,6,7 (MWS process)

   Review of the previous US FDA validation of the WSU MWS process for mashed potatoes revealed the need to confirm that the packaging material used to contain the product did not adulterate it by leaching migrating chemicals in to food. *Risk mitigation:* Printpack has double-sided migration studies of the pouch materials for MWS in last year's chicken and dumplings and this year's Salmon & Alfredo sauce using the US FDA "Chemistry Guidelines" for its "Food Contact Material Notification" program (see ANNEX B).

   The WSU MWS processes for mashed potatoes and salmon & Alfredo defined acceptable methods for inoculating the product with appropriately thermal resistant- spores cold spot of the tray and pouched packages. Identifying the cold spot in a pouch of heterogeneous components is a similar process, each of the elements of the diverse chicken and dumpling food product has its distinct thermal heating properties. MWS process validation effort has identified the dielectric properties of its component foods. Next steps involved determining the package cold spot using a model food and developing the techniques for heat resistant spore inoculation. *Risk mitigation:* WSU personnel replaced two unreliable microwave generators on its pilot line and will begin to recalibrate the modified line to the original one in October 2010. They expect to complete cold spot determination in November. Monthly visits to the Pilot plant are planned to monitor activity and insure progress.

   Tasks 8,9 (Shelf life Modeling) The M-Rule Container Performance Model for Foods has been developed and validated foods and packaging materials on less complex than those involved in shelf stable combat rations. Its adoption to these systems requires an initial calibration of its calculations to the steady state conditions of the material's barrier performance (Transmission rates). *Risk mitigation:* Printpack will use the Model's developer to adapt materials characteristic inputs for these complex non-foil barrier laminations.

5. Tasks 10,11 (TTI Technology)

   TASK 10 (Immediate Solutions): (None… task complete)

   TASK 11 (Intermediate Solutions)

   The time temperature indicator system used to print an indicator message on packaging material must resist not only premature development of indicia during package material conversion, material shipment and storage,

product packaging and sealing but also early message fade during post-processing shipment and storage. The formulation with its TTI functionality must be acceptable in Printpack's printing presses. *Risk mitigation:* Printpack and Segan complete the evaluation of three Segan chemistries in label form in WSU pilot plant runs. Segan prepared selected chemistries in Printpack's thermally-resistant ink vehicle and Segan developed a bench-top lamination simulation to anticipate their printing and laminating compatibility of this formulation must be acceptable in a commercial printing process:

Program Risks

1. Task 1 (Laminations): (None…this task is complete)
2. Task 2 (Physical, Barrier, Optical Data): (None…this task is complete)
3. Task 3 (Photodegradation Data): (None…this task is complete)
4. Tasks 4,5,6,7 (Packaged Products): Printpack's packaging material is now available, but WSU faculty and staff have been focused on developing and submitting documentation for a salmon in Alfredo sauce product. Inoculated pouches of this product are now undergoing incubation. *WSU administration has committed to maintain the WSU MWS process pilot plant at its present location through the end of December, 2010, but they have requested a no-cost contract extension through the end of FY2011. The request to modify the DOD/Printpack contract is currently under review.*
5. Tasks 8 & 9 (Shelf life Modeling): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.
6. Tasks 10 &11 (TTI Technology):

   TASK 10 (Immediate Solutions): (None… task complete)

   TASK 11 (Intermediate Solutions): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.

Issues

Unexpected Issues

TASK AREA	COMMENT
1. Task 1 (Laminations):	(none)
2. Task 2 (Barrier data):	(none)
3. Task 3 (Photodegradation Data)	(none)
4. Tasks 4,6,7 (MWS process)*	• Qualitative study of extractables from unprocessed, MWS processed, and retorted pouches indicates no significant concerns, but has lead to refinement of future protocols for US FDA food contact material compliance. (Annex A) • Principle investigator visited the WSU pilot Plant and lab in early November to assess new microwave generator installation. Calibration of new generators with the water load was completed, but an asymmetrical cold spot location in the horizontal plane is now observed. Trouble shooting is ongoing. • FDA has requested new studies on the z value of the PA 3697 inoculums used for the salmon/Alfredo filing. These should be complete by the end of January.
5. Tasks 8,9 (Shelf life Modeling)	(none)
6. Tasks 10,11 (TTI Technology)	(none)

* Remaining routine (expected) tasks:
    1. determine cold spot
    2. confirm cold spot,
    3. conduct heat penetration tests,
    4. determine processing schedules for inoculated pack studies, and
    5. detect the z-value of spores in chicken breast

Inoculated pack runs should be scheduled in mid-late February.

Details

Good News

TASK AREA	COMMENT
1. Task 1 (Laminations):	• *New improved WVTR films from Toppan (GL-ARHF) provided expected improved results.** • *OTR performance at or below NSRDEC targets from last year duplicated this year**
2. Task 2 (Barrier data): 3. Task 3 (Photodegradation Data)	• *Photodegradation data indicates that 2 layers of pigmented adhesive is sufficient to protect lipids from photooxidation**
4. Tasks 4,6,7 (MWS process)	• *The WSU MWS pilot line will be available for trials throughout all of 2010.** • *Polymeric laminations for food processed produced with improved visual quality and full functionality.** • *Pouches and roll stock for Salmon & Alfredo sauce successfully used for validation trials*.*
5. Tasks 8&9 (Shelflife modeling)	• Significant progress made in calibrating laboratory data for raw materials and polymeric lamination and gathering characteristic food data
6. Task 10 (Immediate TTI Technology)	• *Work essentially complete and successful**
7. Task 11 (Intermediate TTI Technology)	• *Bench top testing of TTI pigment with standard flexographic heat-resistant vehicle confirmed functionality equal to or better than in previous screen printing vehicle** • *Bench top laminations successfully processed through WSU MWS process. **

** Previously reported*

Details

Technical

TASK AREA	COMMENT
1. Task 1 (Laminations):	• Laminations complete (previously submitted).
2. Task 2 (Barrier data):	• Barrier data is summarized in ANNEX A.
3. Task 3 (Photodegradation Data)	• Photodegradation Data complete (previously submitted). • Other physical data is complete for the 10 laminations (submitted with ANNEX A). • WSU has completed its thin film MW resonance testing on the 10 laminations (previously submitted)
4. Tasks 4,6,7 (MWS process)	• The WSU MWS process has received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding. • The WSU MWS process has submitted a validation Report for Salmon & Alfredo using Printpack pouches. (The process previously received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding) • Printpack has successfully laminated all polymeric high barrier structures for both shelf-stable thermally-processed food and hot fill items. WSU is behind schedule with developing engineering data and inoculation methodology.
5. Tasks 8,9 (Shelf life Modeling)	• Barrier performance for laminations and raw material base films will be validated in model by model developer.
8. Task 10 (Immediate TTI Technology)	• Final report with successful results previously submitted.
9. Tasks 11 (Intermediate TTI Technology)	• In depth evaluations of precise and repeated color indications finished on bench top and onto the WSU Pilot Plant for additional confirmation. • Detailed preparations for press run are now in process at both Printpack and Segan.

Details

Financial

Project Expenses as of 9 Dec 2010

COST ELEMENT	Contract	Q-1 Amt	Q-2 Amt	Q-3 Amt	Q-4 Amt	Q-5 Amt	to Date
Total Direct Labor	232,930	28,496	24,112	34,396	6,635	7,438	101,077
Payroll Tax/Benefits	84,511	10,339	8,748	12,497	2,389	2,415	36,388
Dept Overhead	135,996	16,637	14,077	20,081	3,849	3,981	58,625
Labor Total	453,437	55,472	46,938	66,974	12,873	13,833	196,090
Consulting/Services	544,500	0	941	87,909	68,441	217,532	374,823
Mtls/Plant Costs	70,850	33,170	-	16,153	158,940	0	208,263
Travel	24,530	6,660	4,152	11,705	1,980	900	25,397
Other Direct Costs	160,760	99,505	-6,807*	19,144	0	0	118649
Total Costs	1,254,077	194,807	45,224	201,858	229,361	232,265	903,515
10% Fee	125,409	19,481	4,522	20,186	22,936	232,265	299,390
Contract Total	1,379,486	214,288	49,746	222,044	252,297	464,530	1,202,905

Project expenses to date as captured in Printpack's financial accounting system indicate that the work remains on budget. Some of the budgeted direct labor costs are reported here as plant costs. Most of the remaining work will be conducted by the subcontractors.

Equipment*

(No change from last report)

Equipment	Cost	Assoc. Task(s)	Location
OXTRAN® 2/21 SL Env. Chamber Operating. System	$61,641	2	Printpack Analytical Services Lab 5 Barber Industrial Ct. Villa Rica, GA 30180
Leap Autosampler System	$36,107.	3	

* No additional equipment purchases during 2nd, 3rd , 4th or 5th quarters

SUBCONTRACTS

Subcontracts

Subcontractor	Cost	Assoc. Task(s)	Status
Washington State University	$400,533	4,6,7	Work underway; no-cost time extension negotiated with WSU and proposed to DOD
Segan Industries	$144,000.	10.11	Phase 1 complete; work on Phase II underway; on budget. Timing delayed by availability of WSU pilot plant

Amended timing for WSU deliverables as negotiated with Printpack and proposed to DOD is…

Deliverables	Delivery Date
1. Report on thermal stability of selected TTI label materials in high temperature environment suitable for thermal sterilization applications. We may use WSU package film test cells and oil baths to conduct heating tests. Samples will be evaluated at WSU and in Printpack. (See ANNEX C)	Oct 31,2010
2. Report on interaction among microwaves, food package films and foods; with evaluation of thermal stability of Printpack films using 40 kW 915 MHz microwave sterilization system at WSU. Send processed films to Printpack for quality evaluation. Formulation of the standard test food model will be developed in cooperation with Printpack. (See ANNEX D)	Oct 31,2010
3. Developed and validated thermal processing procedures for two food products (chicken dumpling and salmon in Alfredo sauce) in three selected film materials using the 915 MHz microwave sterilization system.	June 30,2011
4. Produce food products in pouches, conduct microbial check, and send 40 pouches of each product to Natick for sensory and shelf-life studies. We will also process the same foods in MRE foil pouches using conventional retorting method for comparison.	Mar 31,2011
5. Assistance to Printpack in studies of commercial scale-up abilities.	Jul 31,2011
6. Assistance to Printpack in testing TTI system for package materials compatible with microwave sterilization system.	Nov 30,2010

ANNEX A

Report on Qualitative Extraction study: MWS-processed pouches

Materials

Printpack supplied the test samples, which consisted of treated pouches (Microwave "09-M" and Retort "09-R"). The sponsor also supplied control samples (untreated pouches-"09-C"). The test and control samples were stored under ambient conditions prior to testing. Information on the purity and stability of the test and control articles under ambient conditions and testing conditions was the responsibility of the sponsor.

The control pouches were unused and unmarked. Prior to test submittal, Printpack treated the "test" pouches. The microwave and retort pouches were filled with Alfredo sauce and a salmon patty or a whey protein gel block (salmon patty model food). Both were thermally treated to raise the internal temperature of the contents to about 121°C. The 09-M series used microwave energy (3 minutes) and were held at sterilizing temperature for 6 minutes. The 09-R series used steam heat and were held at sterilizing temperature for 30 minutes. The pouches were rinsed with DI water to remove any remaining food particles prior to testing.

The food simulant, according to US FDA Guidelines for polyolefin extraction testing, was 95% ethanol (KJB19F, Pharmco-Aaper of Brookfield, CT).

Methods

The two-sided extraction cells consisted of appropriate enclosures capable of withstanding the high pressures generated by heating the extraction solvent past its boiling points. Two sides of the test articles, 24 in^2, were exposed to 240 ml of extraction solvent. The solvent volume to surface area ratio was 10 ml/ in^2, and no precipitate or cloudiness was initially observed in the extracts. After cooling, there were fine white particulates noted in the Microwave "09-M" and Retort "09-R" extracts.

The test and control samples were extracted in triplicate at 121°C for 2 hours, then 40°C for 240 hours. After 240-hours, the extracts were sampled, scanned and screened for non-volatiles and semi-volatiles. A portion of film representing approximately 10 in.2 (100 ml extract) of test material was placed in a 250 ml round bottom flask and taken to near dryness on a rotary evaporator with a water bath temperature of approximately 35°C. The residue was transferred to a graduated centrifuge tube with multiple rinses of acetonitrile. The acetonitrile was concentrated to 5.0 ml and a portion was filtered through a 0.2 μ Teflon filter. An aliquot of the concentrated extract was analyzed using the GC parameters in Table 1.

In addition, a portion of each pouch, (4 in^2) was also analyzed directly via headspace Gas Chromatography (GC) to qualitatively assess and compare volatile organic compounds in the pouches. The 4 in^2 of test material was placed in a 20 ml headspace vial and capped. The sample is heated for approximately 30 minutes at 80°C, and then a portion of the headspace was analyzed using the GC parameters in Table 2.

ANNEX B

GC/MSD Conditions	Table 1: 95% ethanol from Extraction Cells	Table 2: Headspace from pouch
Column:	J&W DB-5ms (30 m x 0.25 mm)	Restek Rxi-624Sil MS (30 m x 0.25 mm)
Film thickness:	0.25 µ	1.4 µ
Detector:	Mass Selective	Mass Selective
Temperatures:		
Column:	70°C for 1 min, 10°C/min to 220°C, hold for 5 min, 20°C/min to 350°C, 350°C for 5 min	40°C for 5 min, 10°C/min to 130°C, 25°C/min to 300°C, 300°C for 1.2 min
Injector:	250°C	150°C, split 5:1
Detector:	Transfer Line: 350°C Source: 150°C Quadrupole: 230°C	Transfer Line: 300°C Source: 150°C Quadrupole: 230°C
Carrier Flow rate:	1 ml/min (He)	1 ml/min (He)
Injection volume:	1 µl	1,000 µl
MSD Scan Range:	29-400 amu	19-350 amu

Results

Qualitative comparisons of the differences between (1) retort and control and (2) retort and MWS-treated pouches indicate that the only differences are assumed to originate from the food product inside the treated pouches, mainly fatty acids and their ethyl esters.

The results are considered promising, but far from definitive. Future studies will be modified to used pouches filled with FDA-suggested fatty food simulants with vapor pressures lower than 95% ethanol. In addition to food oils, such as corn and olive oil, FDA indicates that HB307 (a mixture of synthetic triglycerides, primarily C10,C12, and C14) and Miglyol 812 (a fractionated coconut oil having a boiling range of 240-270°C and composed of saturated C8 (50-65%) and C10 (30-45%) triglycerides) are acceptable. This will allow actual process condition to substitute for the two-sided extraction cells extracted at 121°C for 2 hours, followed by the ten day in-pouch storage at 40°C for the simulant.

Annex B

Shelf Life Model Calibration

Introduction

The M-RULE® Container Performance Model for Foods operates by integrating the fundamentals of permeant diffusion and solubility through polymeric (organic) materials, permeant vapor-liquid equilibriums, and time-dependent stress-relaxation behaviors with critically evaluated physical data for the component packaging materials. The model accommodates inorganic coatings on standard polymeric materials with a user-supplied "Barrier Improvement Factor (BIF)". Instead of the diffusion and solubility appropriate for polymeric materials, the model calculates mass movement of permeant through such coatings by its inferring its flux through that coating.

Typical technical data available for flexible packaging films provides an oxygen transmission rate (O_2TR) for the material (typically "ASTM D3985 - 05 Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor") The rate is expressed in terms of the volume of oxygen (cubic centimeters) at standard conditions passing through a unit area (1 meter squared) of film with unit thickness[1] (25 microns) over a 24 hour period at specific temperature (°C), humidity (%RH) and partial pressure differential (atmosphere). This is a measure of the steady-state rate of transmission of oxygen gas through the polymeric material plastics. It provides for the determination of (*1*) oxygen gas transmission rate (O_2TR), (*2*) the permeance of the film to oxygen gas (PO_2), and (*3*) oxygen permeability coefficient ($P'O_2$) in the case of homogeneous materials. As such it is a contrived laboratory benchmark useful for inter-material comparisons, but not an effective, predictive tool for shelf life prediction (unless temperature, humidity and the oxygen partial pressure differential for the packaged product are sustained as specified for the steady state testing).

The M-RULE® handling of inorganic barrier coatings simply assumes the mass transport through whatever voids exist in that coating as a function of the delivery of the permeant to the film/coating interface and the ability of whatever lies on the opposite face of the coating to remove the permeant (e.g. absorption by another polymer, dilution in a free atmosphere, etc).

The model's handling of polymeric barrier coatings assumes sequential solubility and diffusion across the two polymers. The partial pressure differential on either side of the coated film determines the sequence of transport, through coating into film or vice versa.

The objective of this analysis is to use known O_2TR values for base and coated films to validate various model inputs about the packaging materials before using these inputs to dynamically model the shelf life of combat rations with M-RULE®.

Method

Inorganic Coatings

[1] In the case of coated film or multilayered materials, this assumption of uniform transport over a unit thickness is not appropriate and the O_2TR is reported per actual thickness.

Technical data on the O_2TR of base films and coated films are typically available. If attention is given to ensure consistency of temperature, humidity, and partial pressure conditions, these data can be used to directly compute BIF values for the coated film:

$$BIF = \frac{O_2TR_{base}}{O_2TR_{ctd}}$$

In effect, a BIF value is an indirect measurement of the integrity of the inorganic coating.

Polymeric Coatings

To model layered composite polymeric structures, the model must be supplied critical characteristics of each component. It then calculates mass transport of oxygen through the coated film using these characteristics both to compute the absorption and diffusion through the respective layers and to define the immediate desorption/adsorption environment at the interface of the layers. With technical data for base and coated films, assumptions about the (usually proprietary) critical components of coatings can be assessed in order to fit the model to the technical data.

Hybrid Coatings

M-RULE® allows a user to define the nature and distribution of clay nanoparticles (inorganic materials) dispersed within a polymeric matrix. This definition controls changes in diffusion of oxygen trough the neat polymer. In this way, a hybrid coated-film can be modeled with various assumptions until the results fit published technical data.

Results

Individual film components

Conditions and barrier values were provided by the suppliers of the materials used in the retort pouch structure. The same conditions were entered into M-RULE® and used to optimize the materials until they matched the known data, as presented below in Table 1.

Table 1: Data Sheet and Modeled barrier values for films

	Data Sheet Values		Model Values		
	O_2TR cc•day/m²	WVTR gm•day/m²	O_2TR cc•day/m²	WVTR gm•day/m²	BIF
@12μ OPET-hybrid	85% RH; 0.4-0.8	50	20°C, 85% RH: 0.717	24.6	66
@15μ OBON-hybrid	85% RH; 0.5	240	0.713	26.1	66
*12μ OPET-Al_2O_3 #1	.62	2.02	0.695	1.01	42.5
*12μ OPET-Al_2O_3 #2	.62	1.09	0.695	1.01	42.5
#12μ OPET-PVdC	12	14	12.2	16.3	2.4
12μ OPET	29.4	38	29.56	35.3	n/a
15μ OBON	0% RH: 47-62	100% RH: 310 - 357	0% RH: 47.08	100% RH: 347.5	n/a

75µ CPP	207.9	4	200.97	1.66	n/a
75µ mPE	546	5	571.83	4.15	n/a
* Inorganic Coating ⁺ Polymeric Coating # Hybrid Coating					

The only major difference between reported values from suppliers and model results is with regards to the hybrid-coated BON, highlighted in yellow. However, when used in the composite structure, this 10-fold difference did not appear to have any impact on the combined model results.

Composite structure

The current structure for the MRE Retort Pouch is constructed as follows:

12µ OPET//12µ OPET-Al_2O_3 #2//15µ OBON-hybrid//75µ CPP

The Retort pouch was run through the model under the same conditions as used for the OTR and WVTR analyses completed as part of Task 2: Physical, Barrier & Optical Data.

Test Type	Conditions	Tested Values	Model Values
OTR	23°C; 0% RH	<0.009 – 0.253 cc·day/m2	0.017 cc·day/m2
OTR	23°C; 90% RH	<0.009 – 0.079 cc·day/m2	0.0165 cc·day/m2
WVTR	37.8°C; 90% RH	0.158 – 0.555 gm·day/m2	0.259 gm·day/m2

The model conditions and results provided a close approximation of the conditions and results achieved through lengthy, expensive barrier property testing. With proper optimization of materials and conditions, the M-Rule system can provide consistent OTR and MVTR data for complex packaging structures.

Storage Conditions

Standard: 27°C – 50% RH – 365 days
27°C – 90% RH – 90 days
38°C – 90% RH – 640 days

Extreme: 27°C – 50% RH – 90 days
38°C – 90% RH – 90 days
49°C – 20% RH – 915 days

The following charts are based on values computed for a 7" high x 5.25" wide (73.5 sq in packaging surface) pouch, filled nominally with 227g of chicken and dumplings. Maximum fill model scenarios were also run, with very little variation in results from those seen at nominal fill.

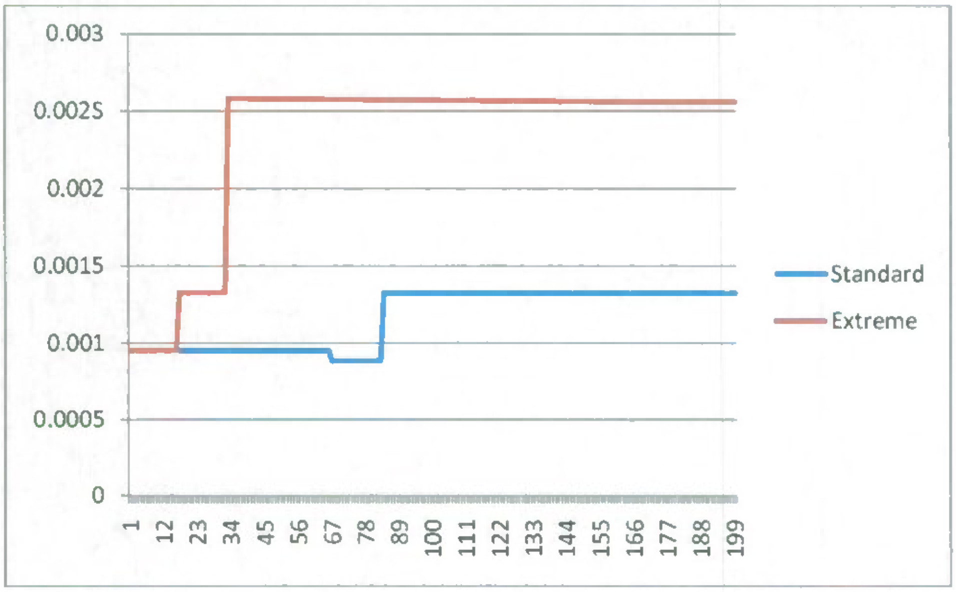


Figure 1. Package Incremental O2 content (cc*day/package) is clearly increased during the extreme storage conditions but remains below 0.003 cc/package per day.

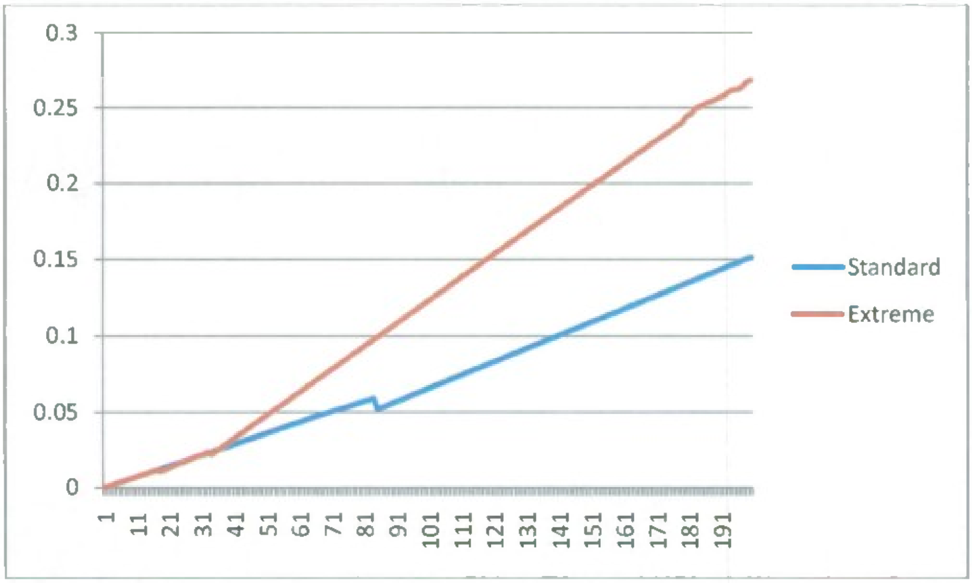


Figure 2. Product O2 content (ppm*day/package) rises throughout the shelf-life but stays relatively low. If particular vitamins or compounds that are oxygen-sensitive could be affected even by such low-level increases, the model can be altered to track vitamin content and activity.

Product Moisture content (%RH/day) was maintained at approximately 76% under both sets of conditions.

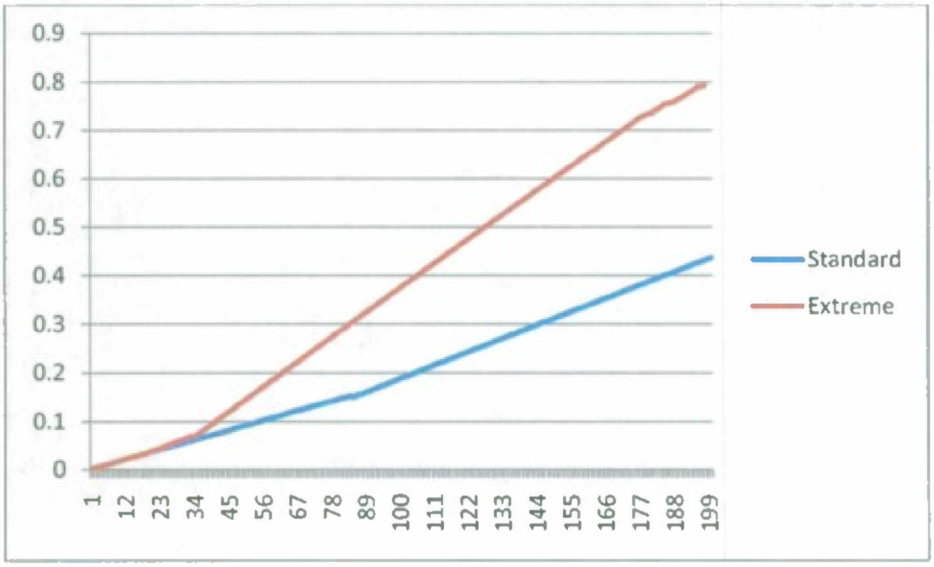


Figure 3. Headspace O2 content (%/day) increases with time, and increases more rapidly under the extreme conditions. As with the product O2 content, this can be monitored more closely over the period of concern and with respect to oxygen-sensitivity.

Headspace Relative Humidity (%/day) was maintained at 100% under both sets of conditions due to the high moisture content of the food.

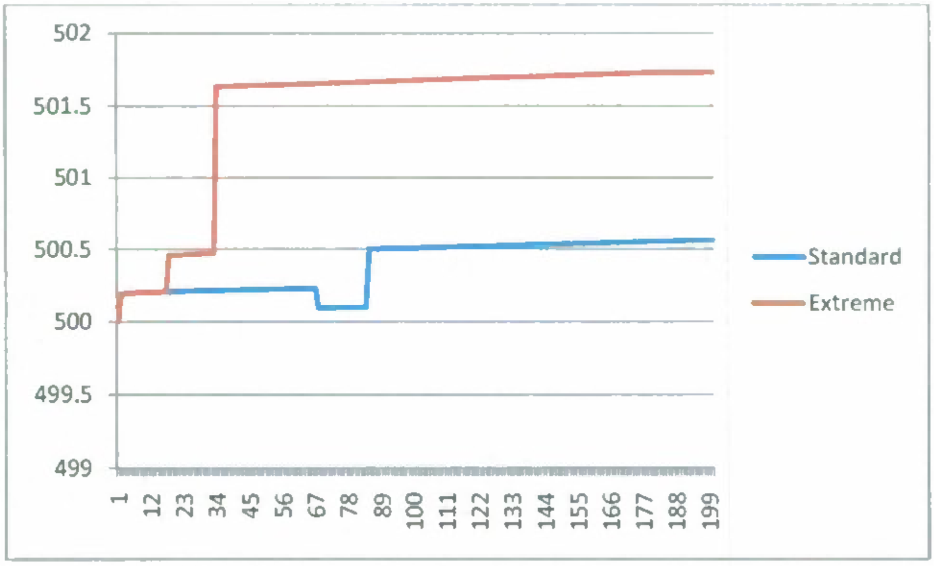


Figure 4. Package Volume (mL/day) started at 500 mL and maintained fairly steadily with a high of 500.6 during standard conditions. During extreme conditions, the package volume swelled to 501.8 mL at the highest.

Draft Quarterly Report

For the Period Ending
31 March, 2011
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Quarterly Report

For the period ending
31 Dec 2010
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Summary: After five quarters, delays at the major subcontractor, Washington State University, have delayed progress for several tasks in this project. Major progress has been made in preparing predictive shelf life models. (Standard and Extreme logistics conditions) and in extended run entrées.

Contents

Project Overview

Objective

To advance the state of the art for Thermal Microwave Sterilization (MWS) for pouches of individual combat rations while maintaining current shelf life:

- improvements in the MWS process at Washington State University (WSU) and
- improvements in the non-foil light barrier materials considered in the Printpack research

Deliverables

Task	Deliverable	Plan date	Act/Ant date
~~1~~	~~10 Laminations~~	~~31 Dec 2009~~	~~08 Jan 2010~~
~~2~~	~~Physical, Barrier, & Optical Data~~	~~28 Feb 2010~~	~~31 Oct 2010~~
~~3~~	~~Photodegradation Data~~	~~31 Jan 2010~~	~~5 Mar 2010~~
4	Retort & MWS Entrée Packages	30 Apr 2010	31 Mar 2011
~~5~~	~~Hot Fill Packages~~	~~30 Apr 2010~~	~~28 Sep 2010~~
6	Optimized MWS Entrée Packages	30 Jun 2010	31 May 2011
7	MWS Validation Report	30 Jun 2010	30 Jun 2011
8	Standard Condition Shelflife Modeling	30 Apr 2010	31 Dec 2010
9	Extreme Condition Shelflife Modeling	31 May 2010	31 Dec 2010
~~10~~	~~TTI Label Evaluation~~	~~30 Apr 2010~~	~~1 July 2010~~
11	Printed Pouch TTI Evaluation	31 Aug 2010	31 Mar 2011

Accomplishments

1. Task 1 (Laminations): All laminations are completed; including advances include new barrier materials and alternate opacifying pigments for sealant films.
2. Task 2 (Material data sheets): Water vapor, oxygen, and light barrier and dielectric and physical properties have been determined for all task 1 laminations.
3. Task 3 (Photodegradation Data): The photodegradation assessment of the 10 sample laminations is completed (GCMS/quantification of hexanal after extended light exposure indicated that several techniques for imparting light barrier functionality to pouches are feasible).
4. Tasks 4, 6, 7 (MWS process): Submitting a USDA validation report for chicken and dumplings at the end of the second quarter of CY2011 is now planned. WSU submitted its FDA validation report for a pouched Salmon & Alfredo sauce item and it has been deemed acceptable. Extended run pouches have been processed and inoculation studies are underway.
5. Tasks 8 &9 (Shelf life Modeling)*: Assistance from the author of the M-Rule food shelf model allowed calibration of the barrier transmission rates of the base films and composite lamination. Data input for chicken and dumplings entrée is now finished. A report has been drafted and is undergoing revisions to ensure that all aspects of the deliverable are addressed thoroughly.
6. Tasks 10 & 11 (TTI Technology): The majority of the lamination is completed. Some setbacks were experienced regarding printing the TTI ink but it has been resolved. Print is scheduled for April. MWS evaluation depends on WSU schedule but should follow shortly.

Risks

Technical and program Risks

Technical Risks

1. Task 1 (Laminations): (None… task complete)
2. Task 2 (None… task complete):
3. Task 3 (Photodegradation Data) : (None… task complete)
4. Tasks 4,6,7 (MWS process)

   Review of the previous US FDA validation of the WSU MWS process for mashed potatoes revealed the need to confirm that the packaging material used to contain the product did not adulterate it by leaching migrating chemicals in to food. *Risk mitigation:* Printpack has double-sided migration studies of the pouch materials for MWS in last year's chicken and dumplings and this year's Salmon & Alfredo sauce using the US FDA "Chemistry Guidelines" for its "Food Contact Material Notification" program (see ANNEX B).

   The WSU MWS processes for mashed potatoes and salmon & Alfredo defined acceptable methods for inoculating the product with appropriately thermal resistant- spores cold spot of the tray and pouched packages. Identifying the cold spot in a pouch of heterogeneous components is a similar process, each of the elements of the diverse chicken and dumpling food product has its distinct thermal heating properties. MWS process validation effort has identified the dielectric properties of its component foods. Next steps involved determining the package cold spot using a model food and developing the techniques for heat resistant spore inoculation.

   Risk mitigation: WSU personnel replaced two unreliable microwave generators on its pilot line and recalibrated the modified line to the original one in October 2010. They completed cold spot and progressed to extended run trials as well as inoculated package studies. The results should be forthcoming by the middle of May.

5. Tasks 8,9 (Shelf life Modeling)
   The M-Rule Container Performance Model for Foods has been developed and validated foods and packaging materials on less complex than those involved in shelf stable combat rations. Its adoption to these systems requires an initial calibration of its calculations to the steady state conditions of the material's barrier performance (transmission rates). The current foil lamination had to be input as well, leading to some additional delays.

   Risk mitigation: Printpack will use the model's developer to adapt materials characteristic inputs for these complex non-foil barrier laminations.

6. Tasks 10,11 (TTI Technology)

   TASK 10 (Immediate Solutions): (None… task complete)

   TASK 11 (Intermediate Solutions)

Risks

The time temperature indicator system used to print an indicator message on packaging material must resist not only premature development of indicia during package material conversion, material shipment and storage, product packaging and sealing but also early message fade during post-processing shipment and storage. The formulation with its TTI functionality must be acceptable in Printpack's printing presses. Attempts to mimic full-scale press operations with a combination of printing on a press and hand printing experienced some set-backs due to lack of clarity in the required order of inks.

Risk mitigation: Printpack and Segan completed the evaluation of three Segan chemistries in label form in WSU pilot plant runs. Segan prepared selected chemistries in Printpack's thermally-resistant ink vehicle and Segan developed a bench-top lamination simulation to anticipate their printing and laminating compatibility of this formulation must be acceptable in a commercial printing process. Confusion over the printing order of inks required for a functioning TTI was clarified and material will be printed shortly.

Program Risks

1. Task 1 (Laminations): (None…this task is complete)
2. Task 2 (Physical, Barrier, Optical Data): (None…this task is complete)
3. Task 3 (Photodegradation Data): (None…this task is complete)
4. Tasks 4,5,6,7 (Packaged Products): WSU has recently been informed that chicken & dumplings entrée requires USDA approval rather than FDA approval. Final approval could be delayed until USDA has undergone training to familiarize the required staff with the technology. The report on the filing should be delivered on time.
5. Tasks 8 & 9 (Shelf life Modeling): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.
6. Tasks 10 &11 (TTI Technology):

   TASK 10 (Immediate Solutions): (None… task complete)

   TASK 11 (Intermediate Solutions): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.

Issues

Unexpected Issues

TASK AREA	COMMENT
1. Task 1 (Laminations):	(none)
2. Task 2 (Barrier data):	(none)
3. Task 3 (Photodegradation Data)	(none)
4. Tasks 4,6,7 (MWS process)*	• Qualitative study of extractables from unprocessed, MWS processed, and retorted pouches indicates no significant concerns, but has lead to refinement of future protocols for US FDA food contact material compliance. (Annex A) • WSU has recently been informed that chicken & dumplings entrée requires USDA approval rather than FDA approval. Final approval could be delayed until USDA has undergone training to familiarize the required staff with the technology. The report on the filing should be delivered on time. • The main contact at WSU, Galina Mikhaylenko, has resigned her position. Frank Liu has taken over her activities but it is possible that some delays may be experienced as changeover occurs.
5. Tasks 8,9 (Shelf life Modeling)	(none)
6. Tasks 10,11 (TTI Technology)	(none)

Details

Good News

TASK AREA	COMMENT
1. Task 1 (Laminations):	• *New improved WVTR films from Toppan (GL-ARHF) provided expected improved results.** • *OTR performance at or below NSRDEC targets from last year duplicated this year**
2. Task 2 (Barrier data): 3. Task 3 (Photodegradation Data)	• *Photodegradation data indicates that 2 layers of pigmented adhesive is sufficient to protect lipids from photooxidation**
4. Tasks 4,6,7 (MWS process)	• *The WSU MWS pilot line will be available for trials throughout all of 2010.** • *Polymeric laminations for food processed produced with improved visual quality and full functionality.** • *Pouches and roll stock for Salmon & Alfredo sauce successfully used for validation trials*.*
5. Tasks 8&9 (Shelflife modeling)	• *Significant progress made in calibrating laboratory data for raw materials and polymeric lamination and gathering characteristic food data** • Foil laminate structure has successfully been approximated in the system
6. Task 10 (Immediate TTI Technology)	• *Work essentially complete and successful**
7. Task 11 (Intermediate TTI Technology)	• *Bench top testing of TTI pigment with standard flexographic heat-resistant vehicle confirmed functionality equal to or better than in previous screen printing vehicle** • *Bench top laminations successfully processed through WSU MWS process. **

** Previously reported*

Details

Technical

TASK AREA	COMMENT
1. Task 1 (Laminations):	▪ Laminations complete (previously submitted).
2. Task 2 (Barrier data):	▪ Barrier data is summarized in ANNEX A.
3. Task 3 (Photodegradation Data)	▪ Photodegradation Data complete (previously submitted). ▪ Other physical data is complete for the 10 laminations (submitted with ANNEX A). ▪ WSU has completed its thin film MW resonance testing on the 10 laminations (previously submitted)
4. Tasks 4,6,7 (MWS process)	▪ The WSU MWS process has received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding and for Salmon & Alfredo using Printpack pouches. ▪ Printpack has successfully laminated all polymeric high barrier structures for both shelf-stable thermally-processed food and hot fill items. ▪ WSU has processed almost all the pouches necessary for the deliverables and has begun inoculated package studies.
5. Tasks 8,9 (Shelf life Modeling)	▪ Barrier performance for laminations and raw material base films have been validated in model by model developer. Full structures have been developed and analyzed using the model system.
8. Task 10 (Immediate TTI Technology)	▪ Final report with successful results previously submitted.
9. Tasks 11 (Intermediate TTI Technology)	▪ In depth evaluations of precise and repeated color indications finished on bench top and onto the WSU Pilot Plant for additional confirmation. ▪ Detailed preparations for press run are now in process at both Printpack and Segan.

Financial

Project Expenses as of 9 Dec 2010

COST ELEMENT	Contract	Q-1 Amt	Q-2 Amt	Q-3 Amt	Q-4 Amt	Q-5 Amt	to Date
Total Direct Labor	232,930	28,496	24,112	34,396	6,635	7,438	101,077
Payroll Tax/Benefits	84,511	10,339	8,748	12,497	2,389	2,415	36,388
Dept Overhead	135,996	16,637	14,077	20,081	3,849	3,981	58,625
Labor Total	453,437	55,472	46,938	66,974	12,873	13,833	196,090
Consulting/Services	544,500	0	941	87,909	68,441	217,532	374,823
Mtls/Plant Costs	70,850	33,170	-	16,153	158,940	0	208,263
Travel	24,530	6,660	4,152	11,705	1,980	900	25,397
Other Direct Costs	160,760	99,505	-6,807*	19,144	0	0	118649
Total Costs	1,254,077	194,807	45,224	201,858	229,361	232,265	903,515
10% Fee	125,409	19,481	4,522	20,186	22,936	232,265	299,390
Contract Total	1,379,486	214,288	49,746	222,044	252,297	464,530	1,202,905

Project expenses to date as captured in Printpack's financial accounting system indicate that the work remains on budget. Some of the budgeted direct labor costs are reported here as plant costs. Most of the remaining work will be conducted by the subcontractors.

Updated financial information through March will be forthcoming soon.

Equipment*

(No change from last report)

Equipment	Cost	Assoc. Task(s)	Location
OXTRAN® 2/21 SL Env. Chamber Operating. System	$61,641	2	Printpack Analytical Services Lab 5 Barber Industrial Ct. Villa Rica, GA 30180
Leap Autosampler System	$36,107.	3	

* No additional equipment purchases during 2nd, 3rd, 4th or 5th quarters

SUBCONTRACTS

Subcontracts

Subcontractor	Cost	Assoc. Task(s)	Status
Washington State University	$400,533	4,6,7	Work underway; no-cost time extension negotiated with WSU and proposed to DOD
Segan Industries	$144,000.	10.11	Phase 1 complete; work on Phase II underway; on budget. Timing delayed by availability of WSU pilot plant and confusion over printing sequence and requirements

Amended timing for WSU deliverables as negotiated with Printpack and proposed to DOD is…

Deliverables	Delivery Date
1. Report on thermal stability of selected TTI label materials in high temperature environment suitable for thermal sterilization applications. We may use WSU package film test cells and oil baths to conduct heating tests. Samples will be evaluated at WSU and in Printpack. (See ANNEX C)	Oct 31,2010
2. Report on interaction among microwaves, food package films and foods; with evaluation of thermal stability of Printpack films using 40 kW 915 MHz microwave sterilization system at WSU. Send processed films to Printpack for quality evaluation. Formulation of the standard test food model will be developed in cooperation with Printpack. (See ANNEX D)	Oct 31,2010
3. Developed and validated thermal processing procedures for two food products (chicken dumpling and salmon in Alfredo sauce) in three selected film materials using the 915 MHz microwave sterilization system.	June 30,2011
4. Produce food products in pouches, conduct microbial check, and send 40 pouches of each product to Natick for sensory and shelf-life studies. We will also process the same foods in MRE foil pouches using conventional retorting method for comparison.	Mar 31,2011
5. Assistance to Printpack in studies of commercial scale-up abilities.	Jul 31,2011
6. Assistance to Printpack in testing TTI system for package materials compatible with microwave sterilization system.	Nov 30,2010

ANNEX A

Report on Qualitative Extraction study: MWS-processed pouches

Materials

Printpack supplied the test samples, which consisted of treated pouches (Microwave "09-M" and Retort "09-R"). The sponsor also supplied control samples (untreated pouches- "09-C"). The test and control samples were stored under ambient conditions prior to testing. Information on the purity and stability of the test and control articles under ambient conditions and testing conditions was the responsibility of the sponsor.

The control pouches were unused and unmarked. Prior to test submittal, Printpack treated the "test" pouches. The microwave and retort pouches were filled with Alfredo sauce and a salmon patty or a whey protein gel block (salmon patty model food). Both were thermally treated to raise the internal temperature of the contents to about 121°C. The 09-M series used microwave energy (3 minutes) and were held at sterilizing temperature for 6 minutes. The 09-R series used steam heat and were held at sterilizing temperature for 30 minutes. The pouches were rinsed with DI water to remove any remaining food particles prior to testing.

The food simulant, according to US FDA Guidelines for polyolefin extraction testing, was 95% ethanol (KJB19F, Pharmco-Aaper of Brookfield, CT).

Methods

The two-sided extraction cells consisted of appropriate enclosures capable of withstanding the high pressures generated by heating the extraction solvent past its boiling points. Two sides of the test articles, 24 in^2, were exposed to 240 ml of extraction solvent. The solvent volume to surface area ratio was 10 ml/ in^2, and no precipitate or cloudiness was initially observed in the extracts. After cooling, there were fine white particulates noted in the Microwave "09-M" and Retort "09-R" extracts.

The test and control samples were extracted in triplicate at 121°C for 2 hours, then 40°C for 240 hours. After 240-hours, the extracts were sampled, scanned and screened for non-volatiles and semi-volatiles. A portion of film representing approximately 10 $in._2$ (100 ml extract) of test material was placed in a 250 ml round bottom flask and taken to near dryness on a rotary evaporator with a water bath temperature of approximately 35°C. The residue was transferred to a graduated centrifuge tube with multiple rinses of acetonitrile. The acetonitrile was concentrated to 5.0 ml and a portion was filtered through a 0.2 μ Teflon filter. An aliquot of the concentrated extract was analyzed using the GC parameters in Table 1.

In addition, a portion of each pouch, (4 in^2) was also analyzed directly via headspace Gas Chromatography (GC) to qualitatively assess and compare volatile organic compounds in the pouches. The 4 in^2 of test material was placed in a 20 ml headspace vial and capped. The sample is heated for approximately 30 minutes at 80°C, and then a portion of the headspace was analyzed using the GC parameters in Table 2.

GC/MSD Conditions	Table 1: 95% ethanol from Extraction Cells	Table 2: Headspace from pouch
Column:	J&W DB-5ms (30 m x 0.25 mm)	Restek Rxi-624Sil MS (30 m x 0.25 mm)
Film thickness:	0.25 µ	1.4 µ
Detector:	Mass Selective	Mass Selective
Temperatures:		
Column:	70°C for 1 min, 10°C/min to 220°C, hold for 5 min, 20°C/min to 350°C, 350°C for 5 min	40°C for 5 min, 10°C/min to 130°C, 25°C/min to 300°C, 300°C for 1.2 min
Injector:	250°C	150°C, split 5:1
Detector:	Transfer Line: 350°C Source: 150°C Quadrupole: 230°C	Transfer Line: 300°C Source: 150°C Quadrupole: 230°C
Carrier Flow rate:	1 ml/min (He)	1 ml/min (He)
Injection volume:	1 µl	1,000 µl
MSD Scan Range:	29-400 amu	19-350 amu

Results

Qualitative comparisons of the differences between (1) retort and control and (2) retort and MWS-treated pouches indicate that the only differences are assumed to originate from the food product inside the treated pouches, mainly fatty acids and their ethyl esters.

The results are considered promising, but far from definitive. Future studies will be modified to used pouches filled with FDA-suggested fatty food simulants with vapor pressures lower than 95% ethanol. In addition to food oils, such as corn and olive oil, FDA indicates that HB307 (a mixture of synthetic triglycerides, primarily C10,C12, and C14) and Miglyol 812 (a fractionated coconut oil having a boiling range of 240-270°C and composed of saturated C8 (50-65%) and C10 (30-45%) triglycerides) are acceptable. This will allow actual process condition to substitute for the two-sided extraction cells extracted at 121°C for 2 hours, followed by the ten day in-pouch storage at 40°C for the simulant.

ANNEX B

Shelf Life Model Calibration

Introduction

The M-RULE® Container Performance Model for Foods operates by integrating the fundamentals of permeant diffusion and solubility through polymeric (organic) materials, permeant vapor-liquid equilibriums, and time-dependent stress-relaxation behaviors with critically evaluated physical data for the component packaging materials.

Typical technical data available for flexible packaging films provides an oxygen transmission rate (O_2TR) for the material (typically "ASTM D3985 - 05 Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor") The rate is expressed in terms of the volume of oxygen (cubic centimeters at standard conditions) passing through a unit area (1 meter squared) of film with unit thickness[1] (25 microns) over a 24 hour period at specific temperature (°C), humidity (%RH) and partial pressure differential (atmosphere). This is a measure of the steady-state rate of transmission of oxygen gas through the polymeric material plastics. It provides for the determination of (*1*) oxygen gas transmission rate (O_2TR), (*2*) the permeance of the film to oxygen gas (PO_2), and (*3*) oxygen permeability coefficient ($P'O_2$) in the case of homogeneous materials. As such it is a contrived laboratory benchmark useful for inter-material comparisons, but not an effective, predictive tool for shelf life prediction (unless temperature, humidity and the oxygen partial pressure differential for the packaged product are sustained as specified for the steady state testing).

The model accommodates *inorganic coatings* on standard polymeric materials with a user-supplied "Barrier Improvement Factor (BIF)". Instead of the diffusion and solubility appropriate for polymeric materials, the model calculates mass movement of permeant through such coatings by its inferring its flux through voids in the coating. The M-RULE® handling of inorganic barrier coatings simply assumes the mass transport through whatever voids exist in that coating as a function of the delivery of the permeant to the film/coating interface and the ability of whatever lies on the opposite face of the coating to remove the permeant (e.g. absorption by another polymer, dilution in a free atmosphere, etc).

The model's handling of *polymeric barrier coatings* assumes sequential solubility in and diffusion across the boundary of two polymers. The partial pressure differential on either side of the coated film determines the sequence of transport, through coating into film or vice versa.

The objectives of this analysis are to validate various model inputs about the packaging materials with known O_2TR values for base and coated films and then using these inputs to dynamically model the shelf life of combat rations with M-RULE®.

[1] In the case of coated film or multilayered materials, this assumption of uniform transport over a unit thickness is not appropriate and the O_2TR is reported per actual thickness.

Method

Inorganic Coatings

Technical data on the O_2TR of base films and coated films are typically available. If attention is given to ensure consistency of temperature, humidity, and partial pressure conditions, these data can be used to directly compute BIF values for the coated film:

$$BIF = \frac{O_2TR_{base}}{O_2TR_{ctd}}$$

In effect, a BIF value is an indirect measurement of the integrity of the inorganic coating.

Polymeric Coatings

To model layered composite polymeric structures, the model must be supplied critical characteristics of each component. It then calculates mass transport of oxygen through the coated film using these characteristics both to compute the absorption into and diffusion through the respective layers and to define the immediate desorption/adsorption environment at the interface of the layers. With technical data for base and coated films, assumptions about the (usually proprietary) critical components of coatings can be inferred in order to fit the model to the technical data.

Hybrid Coatings

M-RULE® allows a user to define the nature and distribution of clay nanoparticles (inorganic materials) dispersed within a polymeric matrix. This definition controls changes in diffusion of oxygen trough the neat polymer. In this way, a hybrid coated-film can be modeled with various assumptions until the results fit published technical data.

Storage Conditions

The following assumed values for temperature and humidity were modeled using the system. The assumed scenarios represented this logistics sequences:

Scenario	Stage 1	Stage 2	Stage 3
Standard	Domestic warehouse	marine shipment	jungle warehouse
Extreme	Domestic warehouse	marine shipment	desert warehouse

Specifically, these are:

Standard:	27°C – 50% RH – 365 days
	27°C – 90% RH – 90 days
	38°C – 90% RH – 640 days
Extreme:	27°C – 50% RH – 90 days

38°C – 90% RH – 90 days
49°C – 20% RH – 915 days

Results

Individual film components

Conditions and barrier values were provided by the suppliers of the materials used in the MRE non-foil pouch structure. The same conditions were entered into M-RULE® and used to optimize the material characteristics until they matched the known data, as presented below in Table 1.

Table 1: Data Sheet and Modeled barrier values for films					
	Data Sheet Values		Model Values		
	O_2TR cc•day/m²	**WVTR gm•day/m²**	**O_2TR cc•day/m²**	**WVTR gm•day/m²**	**BIF**
@12μ OPET-hybrid	85% RH; 0.4-0.8	50	20°C, 85% RH: 0.717	24.6	66
@15μ OBON-hybrid	85% RH; 0.5	240	0.713	26.1	66
*12μ OPET-Al_2O_3 #1	.62	2.02	0.695	1.01	42.5
*12μ OPET-Al_2O_3 #2	.62	1.09	0.695	1.01	42.5
#12μ OPET-PVdC	12	14	12.2	16.3	2.4
12μ OPET	29.4	38	29.56	35.3	n/a
15μ OBON	0% RH: 47-62	100% RH: 310 - 357	0% RH: 47.08	100% RH: 347.5	n/a
75μ CPP	207.9	4	200.97	1.66	n/a
75μ mPE	546	5	571.83	4.15	n/a

* Inorganic Coating # Polymeric Coating @ Hybrid Coating

The only significant difference between reported values from suppliers and model results is with regards to the hybrid-coated BON, highlighted in yellow. The model inputs could estimate the oxygen barrier of the material very closely, but those same inputs lead to a 10-fold difference in moisture barrier. However, when used in the non-foil composite structure, this 10-fold difference did not appear to have any impact on the results for the composite MRE non-foil pouch, as is shown below.

Composite structure

The current structure for the MRE non-foil pouch is constructed as follows:

12μ OPET//12μ OPET-Al_2O_3 #2//15μ OBON-hybrid//75μ CPP

The MRE non-foil pouch (structure #6) was evaluated using the model under the same conditions entered for the OTR and WVTR analyses completed as part of Task 2: Physical, Barrier & Optical Data.

Test Type	Conditions	Tested Values	Model Values
OTR	23°C; 0% RH	<0.009 – 0.253 cc·day/m2	0.017 cc·day/m2
OTR	23°C; 90% RH	<0.009 – 0.079 cc·day/m2	0.017 cc·day/m2
WVTR	37.8°C; 90% RH	0.158 – 0.555 gm·day/m2	0.259 gm·day/m2

The conditions and characteristics as input into the model provided a close approximation of the conditions and results achieved through lengthy, expensive barrier property testing. With proper optimization of material characteristics and conditions, the M-Rule system can provide consistent OTR and MVTR data for complex packaging structures.

The following charts are based on values computed for a 7.25" high x 5.25" wide (76.125 sq in packaging surface) pouch filled nominally with 227g of chicken and dumplings, a 5.25" high x 3.75" wide (39.375 sq in packaging surface) pouch filled nominally with 40g of peanut butter dessert bar and a 7.375" high x 4.75" wide (73.625 sq in packaging surface) filled nominally with 128g of mango peach applesauce, respectively. Maximum fill model scenarios were also run, with very little variation in results from those seen at nominal fill.

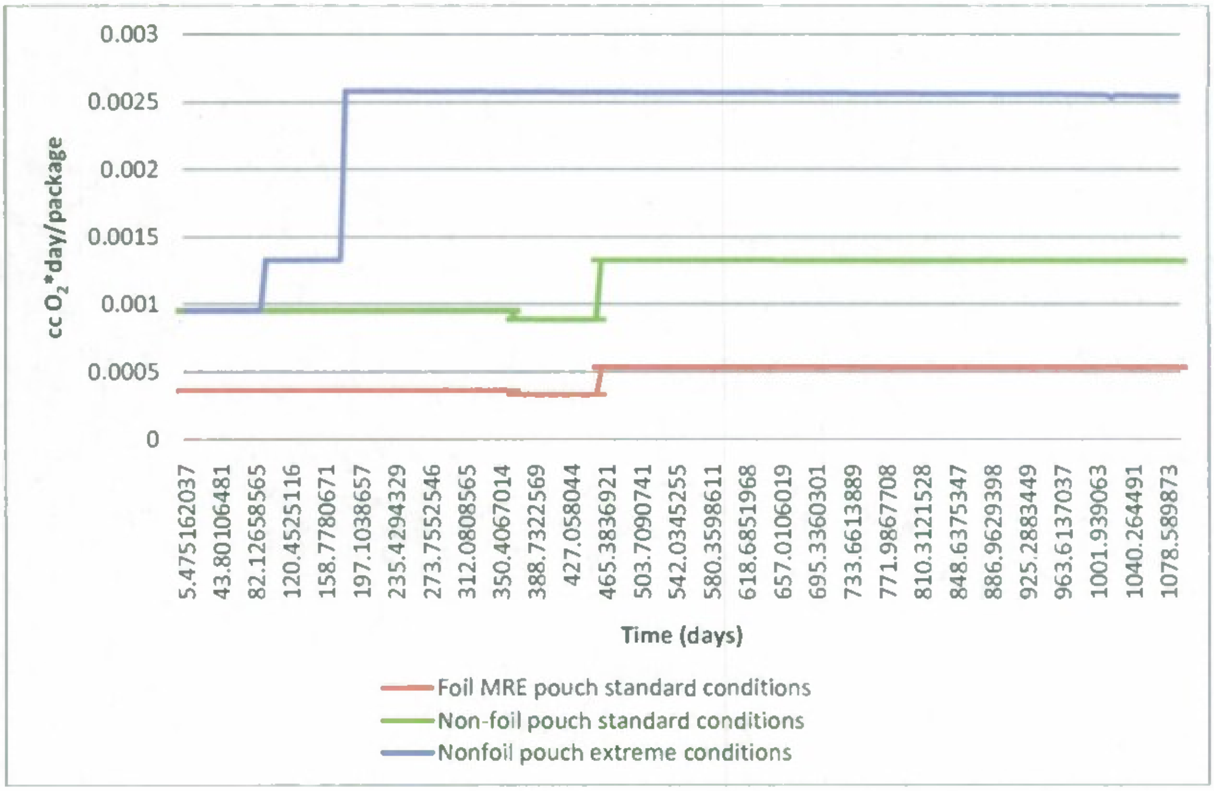


Figure 1. Chicken & Dumplings Package Incremental O_2 content (cc*day/package) is clearly increased during the extreme storage conditions but remains below 0.003 cc*day/package for the non-foil pouch structure. In comparison, foil MRE pouches remain below 0.001 cc*day/package throughout the entire 3 year shelf life of the product under standard storage conditions.

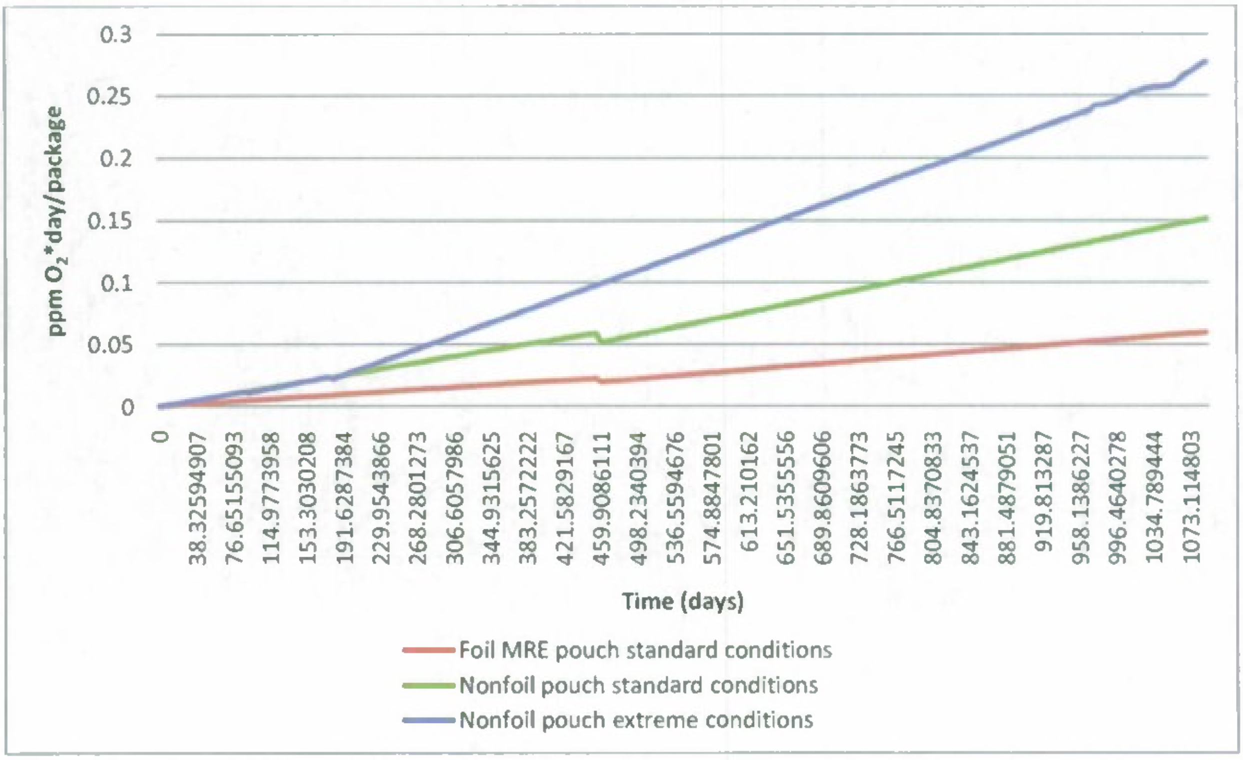


Figure 2. Chicken & Dumplings Product O_2 content (ppm*day/package) rose throughout the shelf-life but stayed relatively low under standard conditions for the non-foil pouch. Under extreme conditions, the product O_2 content nearly doubled from 0.15 ppm *day/package to 0.28 ppm*day/package. If particular vitamins or compounds that are oxygen-sensitive could be affected even by such low-level increases, the model inputs can be altered to track vitamin content and activity.

Chicken & Dumplings Product Moisture content (%RH/day) was maintained at approximately 76% for all package types under both sets of conditions, which used 50% RH as the environmental condition and 0% RH in the headspace gas composition (degassed fill) at filling time. The chicken and dumplings were given a lower moisture specification of 50% RH and an upper moisture specification of 100% RH. If more refined moisture specifications are vital for product quality and regulatory requirements, the current non-foil structure as well as future material changes can be modeled to determine the effect on moisture content as necessary.

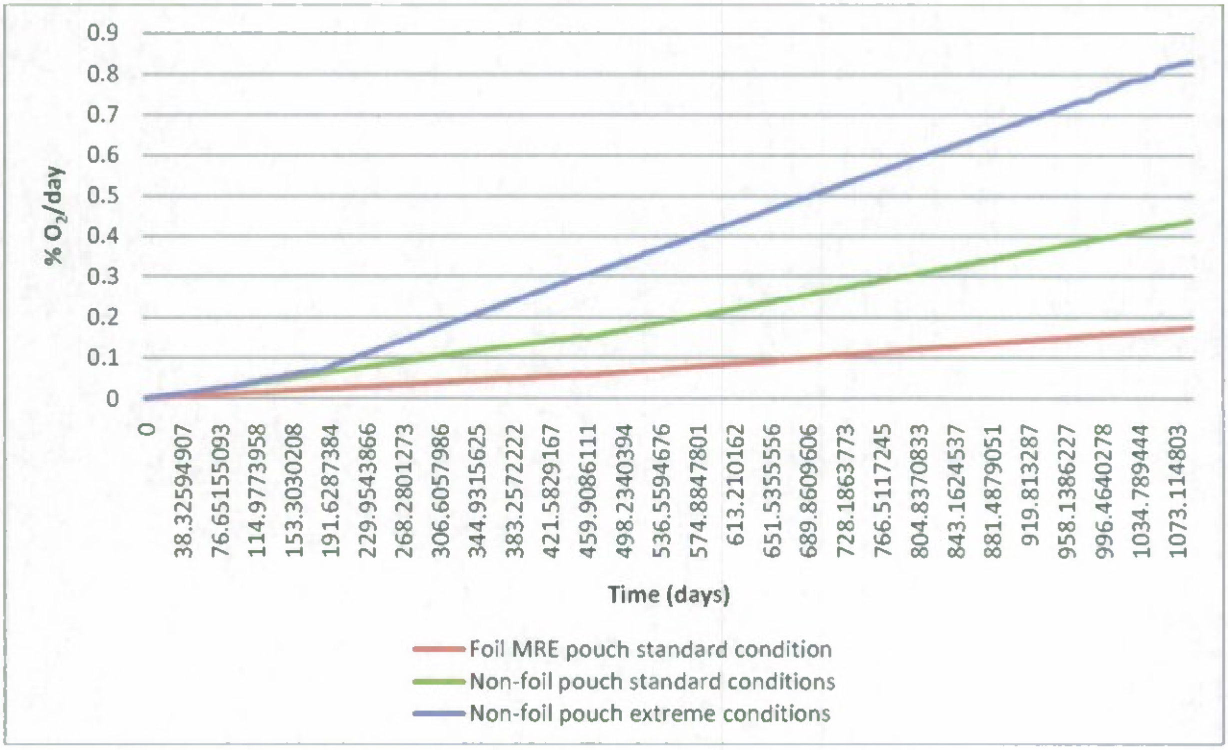


Figure 3. Chicken & Dumplings Headspace O_2 content (%/day) increased with time, and increased more rapidly under the extreme conditions. The foil structure maintained headspace O_2 at below 0.2% over the standard 3 year shelf-life, while the non-foil stayed below 0.45% during the standard shelf-life and below 0.85% during the extreme storage conditions. As with the product O_2 content, this can be monitored more closely over the period of concern and with respect to oxygen-sensitivity.

Chicken & Dumplings Headspace Relative Humidity (%/day) was maintained at 100% for both the foil and non-foil structures under both sets of conditions due to the high moisture content of the food.

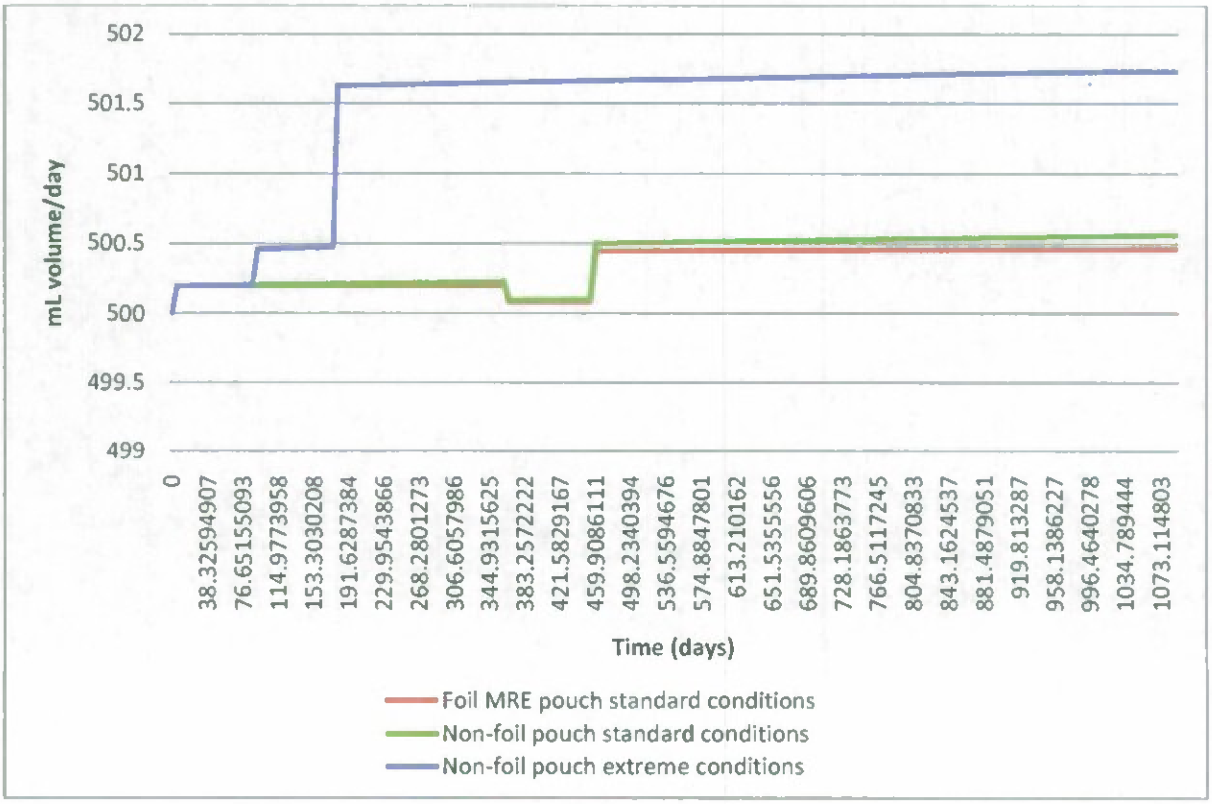


Figure 4. Chicken & Dumplings Package Volume (mL/day) started at 500 mL and maintained fairly steadily with a high of 500.6 during standard conditions. During extreme conditions, the package volume swelled more but stayed under 501.8 mL at the highest. This represents a percent volume increase of 0.12% for the foil and non-foil pouch under standard conditions and a percent volume increase of 0.36% for the non-foil pouch under extreme conditions.

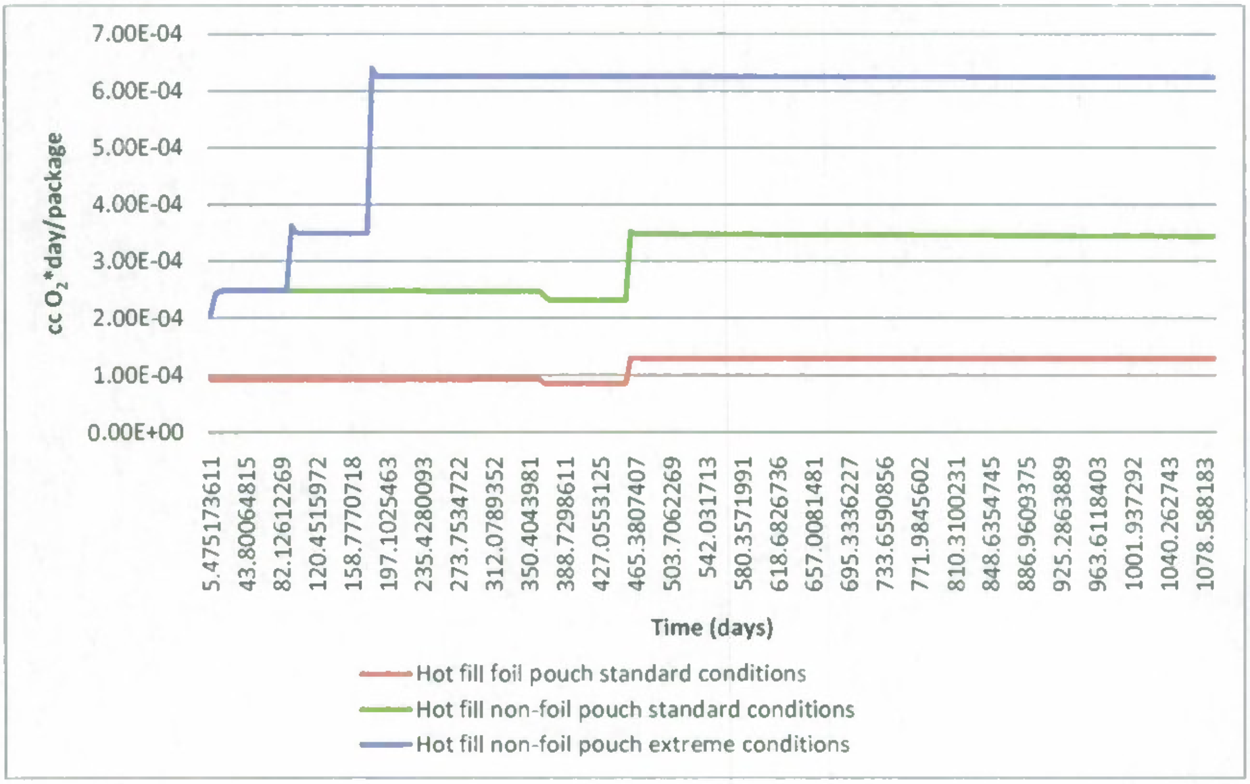


Figure 5. Peanut Butter Bar Package Incremental O_2 content (cc*day/package) was maintained below 0.001 cc*day/package in both structures under standard as well as extreme conditions. The oxygen content was kept lowest in the foil pouch at 0.000128 cc*day/package under standard conditions, whereas the non-foil structure resulted in 0.00034 cc*day/package under standard conditions and 0.00062 cc*day/package under extreme conditions.

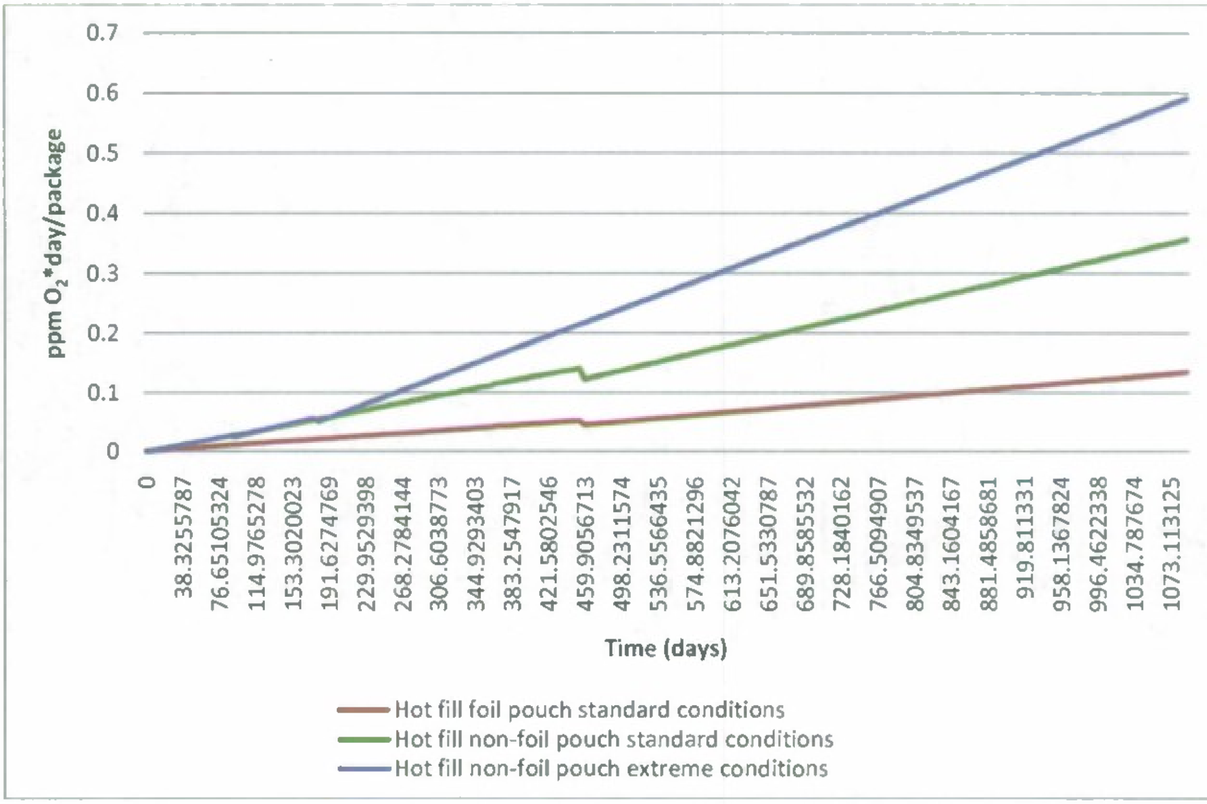


Figure 6. Peanut Butter Bar Product O_2 content (ppm*day/package) increased over the shelf life of the bar to 0.134 ppm*day/package for the foil pouch at standard conditions, 0.356 ppm*day/package for the non-foil pouch at standard conditions and 0.590 ppm*day/package for the non-foil pouch at extreme conditions.

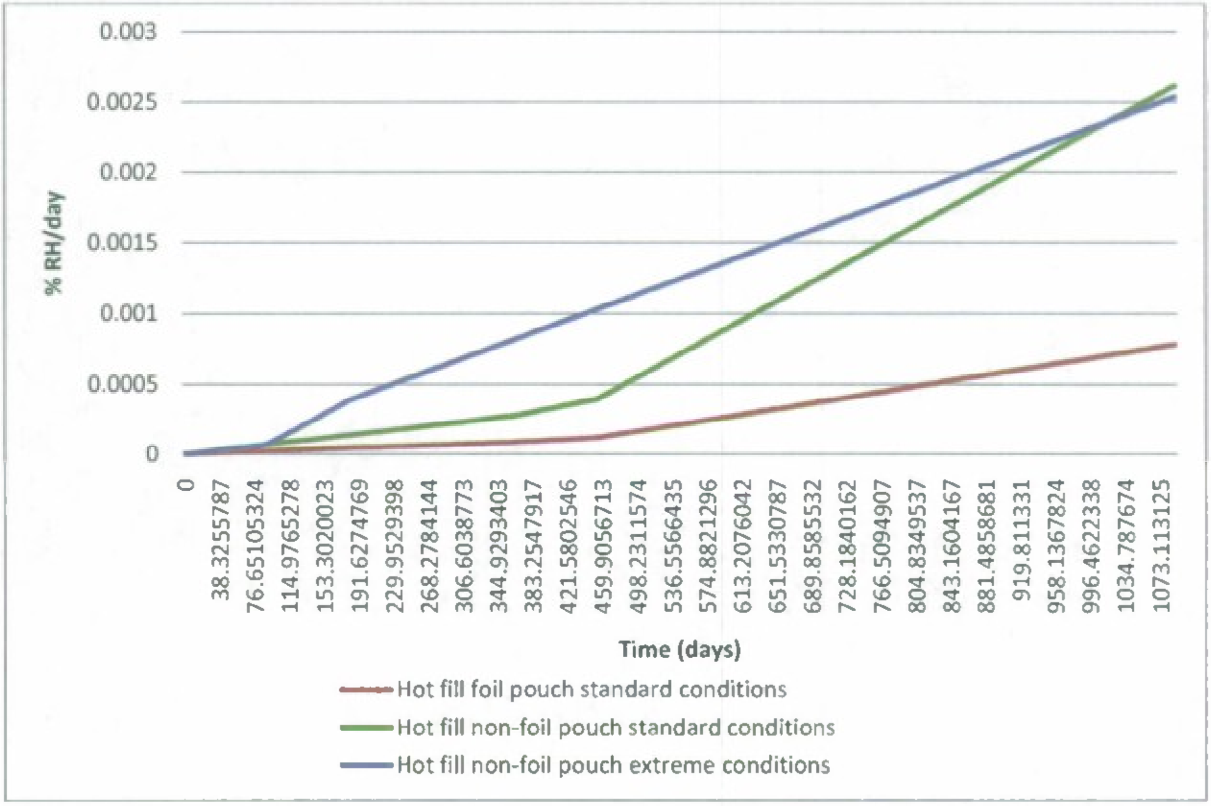


Figure 7. Peanut Butter Bar Product Moisture content (%RH/day) reached 0.0008 %RH/day in the foil pouch over the course of the 3 year shelf life under standard conditions, while storage in the non-foil pouch under standard conditions and extreme conditions yielded a moisture content of 0.0026. This is a significant increase on product moisture content, and while it may still be within the permissible limits, it is obviously an area for improvement in the package capabilities.

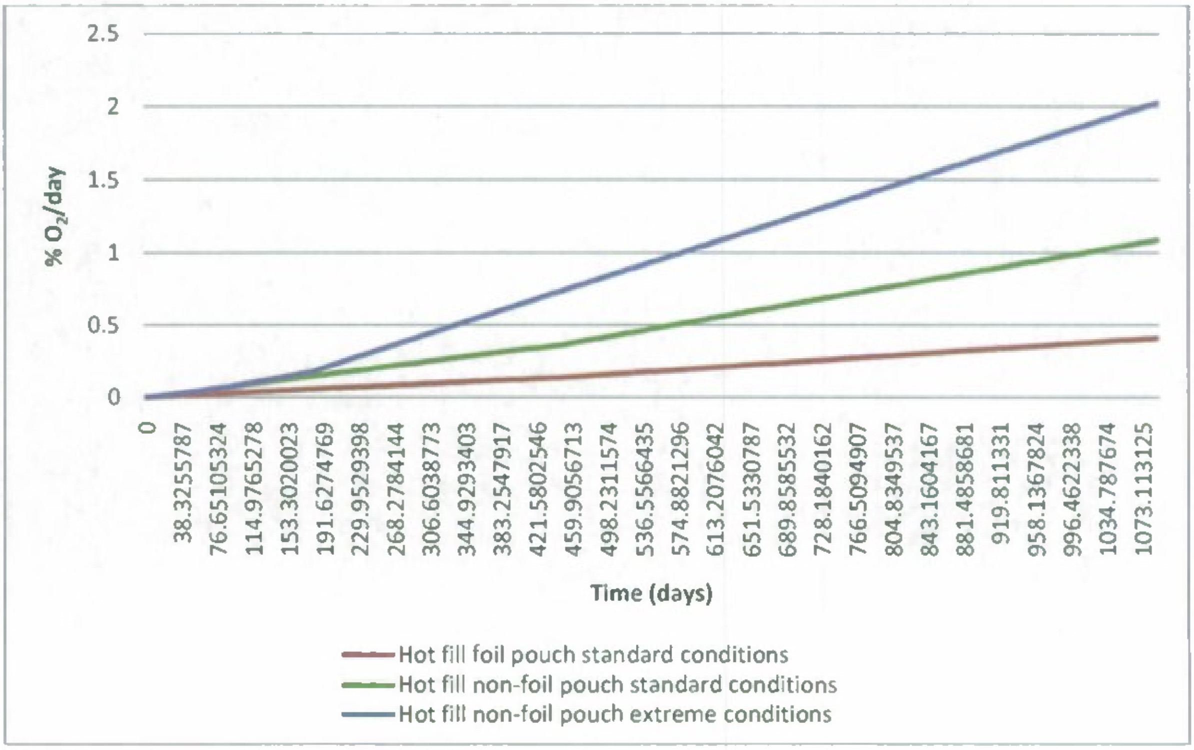


Figure 8. Peanut Butter Bar Headspace O_2 content (%/day) increased more rapidly under the extreme conditions. The foil structure maintained headspace O_2 at below 0.5% over the standard 3 year shelf-life, while the non-foil stayed below 1.1% during the standard shelf-life and at 2% during the extreme storage conditions.

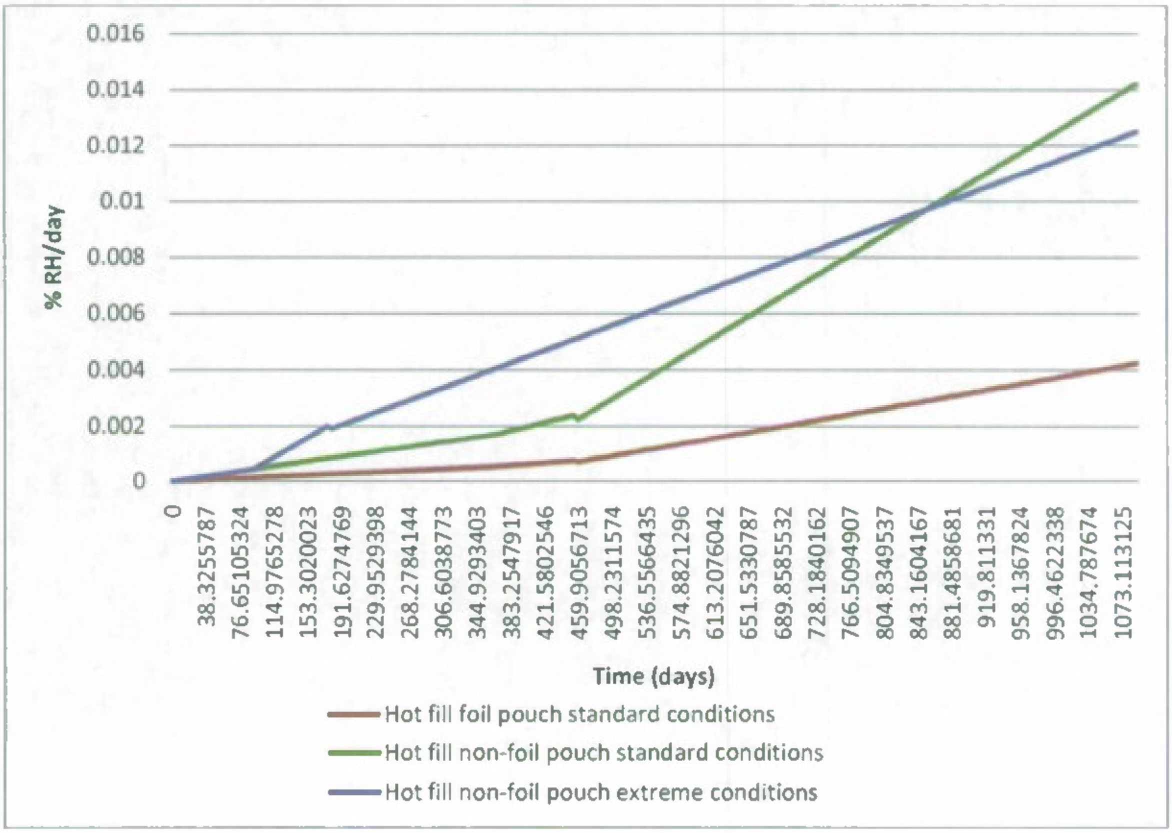


Figure 9. Peanut Butter Bar Headspace Relative Humidity (%/day) reached a high of 0.0042%/day in the foil pouch at standard conditions. The non-foil pouches showed a large increase in headspace RH over the 3 year shelf-life, reaching 0.014 under standard storage conditions and 0.0125 under extreme storage conditions.

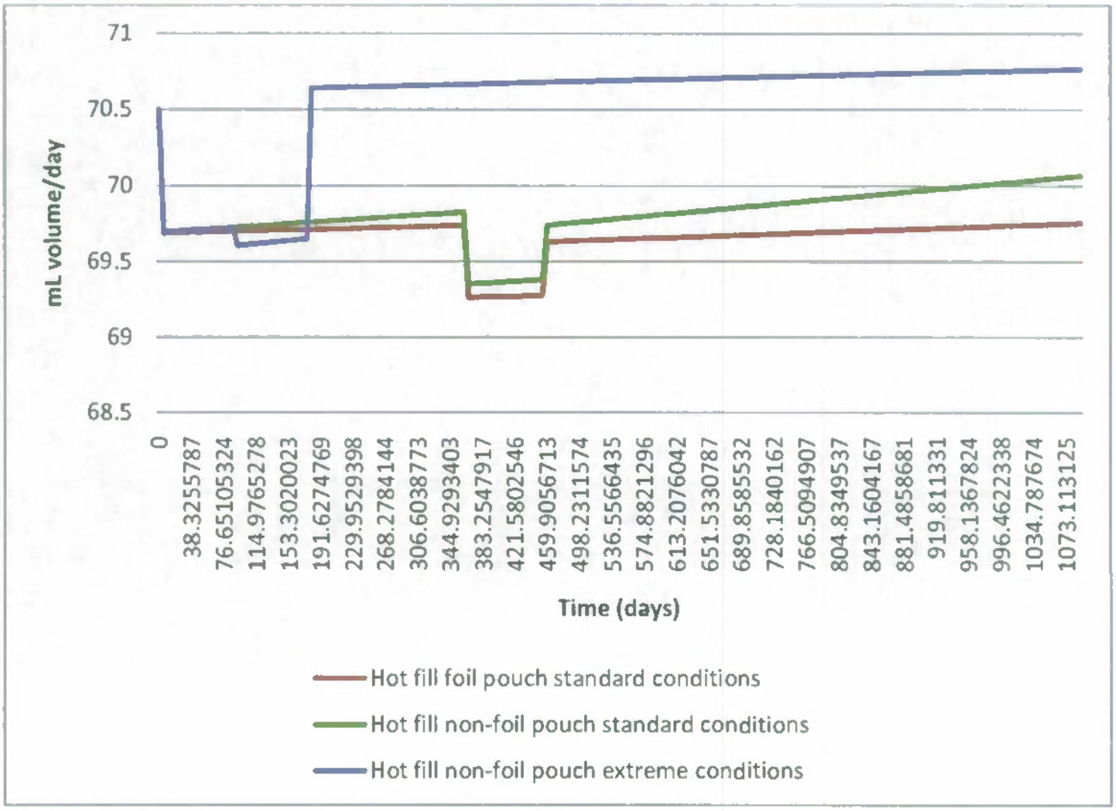


Figure 10. Peanut Butter Bar Package Volume (mL/day) decreased from 70.5 mL to 69.75 in the foil pouch and to 70.07 in the non-foil pouch under standard storage conditions. Volume increased to 70.77 mL in the non-foil pouch under extreme storage conditions. This represents a percent volume decrease of 1.0 % in the foil pouch and 0.6% in the non-foil pouch at standard storage conditions. The non-foil pouch volume increased by 0.38% under extreme storage conditions.

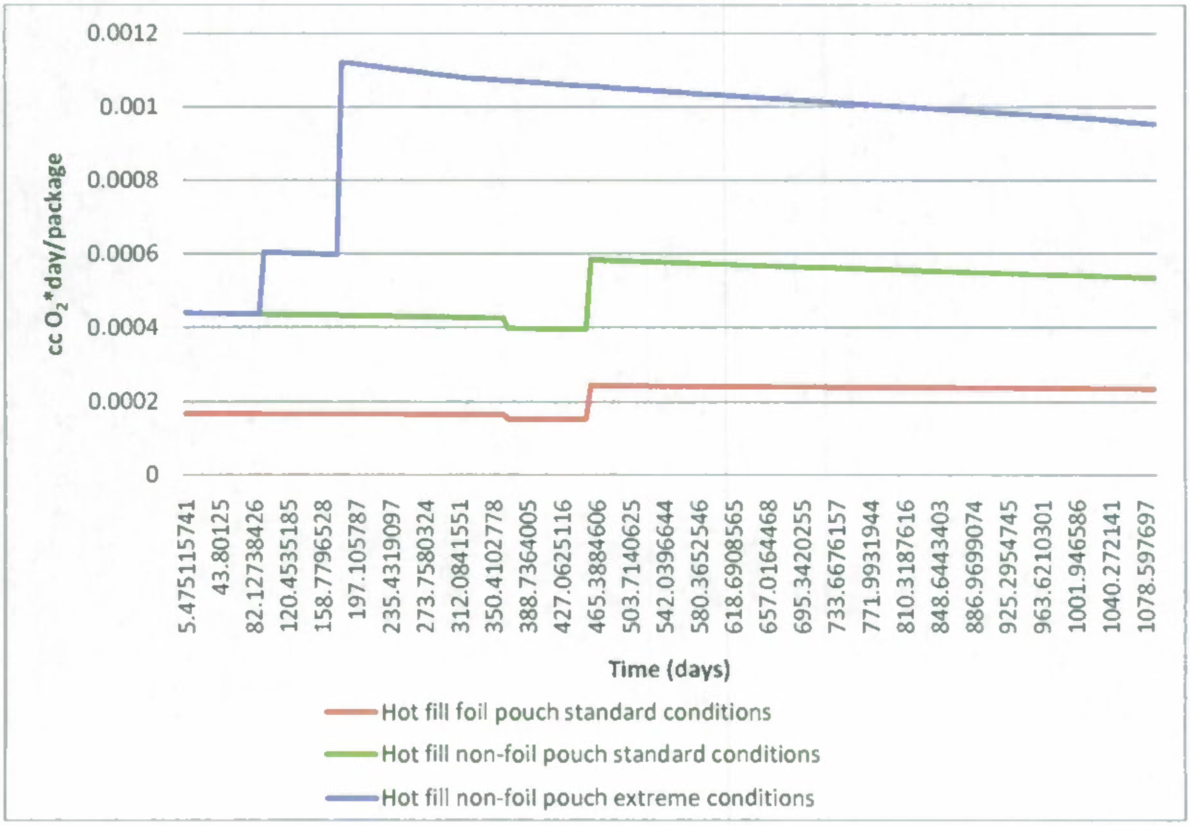


Figure 11. Mango Peach Applesauce Package Incremental O_2 content (cc*day/package) fluctuated over the 3 year shelf life for all samples, settling at approximately 0.0002 cc*day/package for the foil pouch at standard conditions, 0.0005 cc*day/package for the non-foil pouch at standard conditions and 0.00095 cc*day/package for the non-foil pouch at extreme conditions.

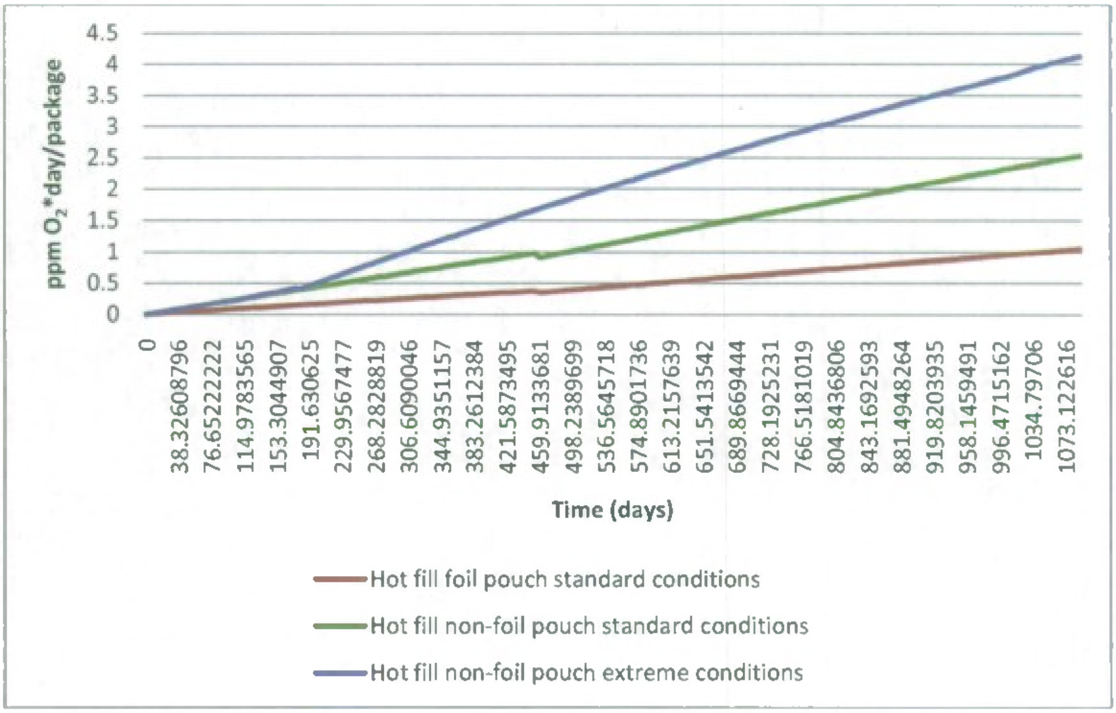


Figure 12. Mango Peach Applesauce Product O_2 content (ppm*day/package) reached 1.04 ppm*day/package in the foil pouches under standard storage conditions, 2.53 ppm*day/package in the non-foil pouches under standard storage conditions and 4.12 in the non-foil pouches under extreme storage conditions.

Mango Peach Applesauce Product Moisture content (%RH/day) was maintained at approximately 77.5% for all package types under both sets of conditions, which used 50% RH as the environmental condition and 0% RH in the headspace gas composition (degassed fill) at filling time. The mango peach applesauce was given a lower moisture specification of 77% RH and an upper moisture specification of 100% RH.

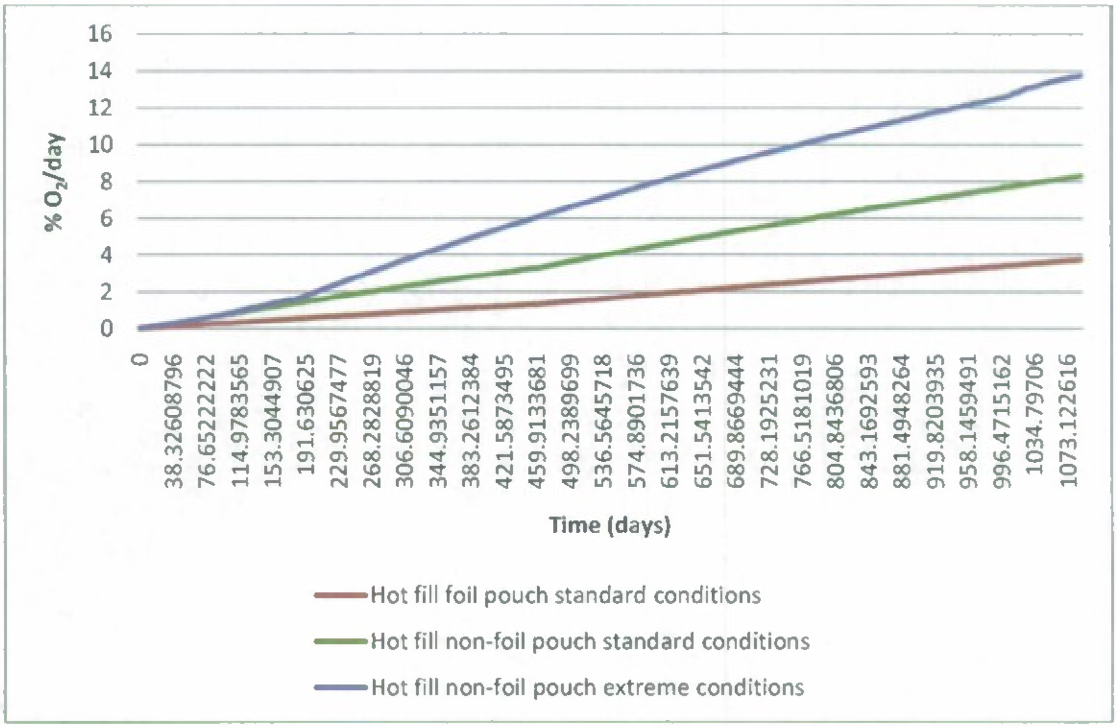


Figure 13. Mango Peach Applesauce Headspace O_2 content (%/day) increased with time, and increased more rapidly under the extreme conditions. The foil structure maintained headspace O_2 at below 4% over the standard 3 year shelf-life, while the non-foil stayed below 8.5% during the standard shelf-life and below 14% during the extreme storage conditions. This is a relatively rapid increase in headspace O_2, even in the foil pouch, and therefore requires additional research.

Mango Peach Applesauce Headspace Relative Humidity (%/day) was maintained at 100% for both the foil and non-foil structures under both sets of conditions due to the high moisture content of the food.

Mango Peach Applesauce Package Volume (mL/day) was maintained at 128 mL in the foil pouch and in the non-foil pouch under standard storage conditions. Volume increased to 128.55 mL in the non-foil pouch under extreme storage conditions. This represents a percent volume increase of 0.43%.

Discussion

Moisture barrier and oxygen barrier are of critical importance when packaging a product that undergoes long-term storage under fluctuating storage conditions, like combat rations. Water activity (also known as the relative vapor pressure of water) is temperature dependant, with the degree of dependence being a function of the moisture content (Fennema, Owen R. (1996). *Food Chemistry, Third Edition*, Marcel Dekker Inc., New York, NY). Water activity can affect multiple areas of food stability, including microbial growth, lipid oxidation and Maillard browning reactions. Oxygen is necessary for many key reactions in food products, including deleterious reactions like lipid oxidation, discoloration (whether by oxidative browning or pigment oxidation) and the growth of aerobic spoilage microorganisms (Eskin, N.A. Michael & Robinson, David S. (2001). *Food Shelf Life Stability: Chemical, Biochemical and Microbiological Changes*, CRC Press LLC, Boca Raton, FL.)

Moisture content and headspace relative humidity for the chicken & dumplings as well as the mango peach applesauce do not appear to undergo extreme variability over the shelf life. The peanut butter bar (moisture content per specification: 0.64 g in a 40 g bar, or 0.016 g H_20/g dry matter) experiences more changes in the moisture content and relative humidity. The current foil MRE pouch structure provides a better barrier than the non-foil pouch structure; however, even in the non-foil pouch during extreme conditions, the moisture content and relative humidity do not appear to increase to unacceptable levels. At low water activity levels, the limiting factor of food acceptability will not be microbial spoilage but lipid oxidation. As the peanut butter bar has 17.16 g of fat in a 40 g bar, as well as fat-soluble vitamins A and E, lipid oxidation is a matter of real concern over the shelf-life of the product.

Oxidative reactions contribute to lipid breakdown, vitamin degradation, phenolic browning and breakdown of color compounds in packaged food products. It is fairly clear from the results for all three foods that the ingress of oxygen is highly dependent on the temperature fluctuations seen over the three year shelf-life. The rate of oxygen ingress through the non-foil package into the headspace and product dramatically increases during the extreme storage scenario, in some cases at the 90 day mark where the conditions change from 27°C/50% RH to 38°C/90% RH, and in others at the 180 day mark of the shelf-life where the conditions change again to 49°C/20% RH for the duration of the shelf-life. However, the oxygen sensitivity can vary based on the proportions and importance of different macronutrients and micronutrients. High-moisture foods like applesauce or chicken & dumplings will not be overly susceptible to lipid or Vitamin A degradation, while the low-moisture, high-fat dessert bar could be greatly impacted by oxidation of various nutrients. However, the high level of Vitamin C in the mango peach applesauce will be susceptible to oxygen degradation as well.

Conclusion

The M-RULE® Container Performance Model for Foods has clearly been shown to validate various model inputs for base and coated films with known O_2TR and MVTR values as well as composite foil and non-foil structures. These inputs have subsequently been used in modeling the anticipated shelf-life of representative combat ration items (an entrée, a fruit sauce and a dessert) over varying storage conditions in the foil and non-foil packages. The results have clarified the progress made in the oxygen and moisture barriers of the non-foil film while pinpointing areas for improvement in future research.

Draft Quarterly Report

For the Period Ending
30 June, 2011
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Quarterly Report

For the period ending
30 June 2011
W911QY-09-C-0205 (FFP)
(Awarded 26 Sep 09)
Printpack Inc.

Summary: Major progress has been made in preparing predictive shelf life models (Standard and Extreme logistics conditions) and in extended run entrées. Inoculated pack studies are wrapping up and Time Temperature Indicator technology has been tested.

Contents

Project Overview

Objective

To advance the state of the art for Thermal Microwave Sterilization (MWS) for pouches of individual combat rations while maintaining current shelf life:

- improvements in the MWS process at Washington State University (WSU) and
- improvements in the non-foil light barrier materials considered in the Printpack research

Deliverables

Task	Deliverable	Plan date	Act/Ant date
~~1~~	~~10 Laminations~~	~~31 Dec 2009~~	~~08 Jan 2010~~
~~2~~	~~Physical, Barrier, & Optical Data~~	~~28 Feb 2010~~	~~31 Oct 2010~~
~~3~~	~~Photodegradation Data~~	~~31 Jan 2010~~	~~5 Mar 2010~~
~~4~~	~~Retort & MWS Entrée Packages~~	~~30 Apr 2010~~	~~31 Mar 2011~~
~~5~~	~~Hot Fill Packages~~	~~30 Apr 2010~~	~~28 Sep 2010~~
6	Optimized MWS Entrée Packages	30 Jun 2010	30 Jun 2011
7	MWS Validation Report	30 Jun 2010	30 Jun 2011
~~8~~	~~Standard Condition Shelflife Modeling~~	~~30 Apr 2010~~	~~31 Mar 2011~~
~~9~~	~~Extreme Condition Shelflife Modeling~~	~~31 May 2010~~	~~31 Mar 2011~~
~~10~~	~~TTI Label Evaluation~~	~~30 Apr 2010~~	~~1 July 2010~~
11	Printed Pouch TTI Evaluation	31 Aug 2010	30 June 2011

Accomplishments

1. Task 1 (Laminations): All laminations are completed; including advances include new barrier materials and alternate opacifying pigments for sealant films.
2. Task 2 (Material data sheets): Water vapor, oxygen, and light barrier and dielectric and physical properties have been determined for all task 1 laminations.
3. Task 3 (Photodegradation Data): The photodegradation assessment of the 10 sample laminations is completed (GCMS/quantification of hexanal after extended light exposure indicated that several techniques for imparting light barrier functionality to pouches are feasible).
4. Tasks 4, 6, 7 (MWS Process): Submitting a USDA validation report for chicken and dumplings at the end of the second quarter of CY2011 is now planned. WSU submitted its FDA validation report for a pouched Salmon & Alfredo sauce item and it has been deemed acceptable. Extended run pouches have been processed and inoculation studies are underway. The 40 pouches intended for sensory study have been shipped to Natick. Progress report is presented in Annex A.
5. Tasks 8 &9 (Shelf Life Modeling): Assistance from the author of the M-Rule food shelf model allowed calibration of the barrier transmission rates of the base films and composite lamination. Data input for chicken and dumplings entrée is now finished. Report has been accepted by Natick.
6. Tasks 10 & 11 (TTI Technology): The lamination is completed. Some setbacks were experienced regarding printing the TTI ink but it has been resolved. MWS evaluation has been completed and pouches have been received at Printpack. Evaluation is underway.

Risks

Technical and program Risks

Technical Risks

1. Task 1 (Laminations): (None… task complete)
2. Task 2 (None… task complete):
3. Task 3 (Photodegradation Data) : (None… task complete)
4. Tasks 4,6,7 (MWS process)

   Review of the previous US FDA validation of the WSU MWS process for mashed potatoes revealed the need to confirm that the packaging material used to contain the product did not adulterate it by leaching migrating chemicals in to food. *Risk mitigation:* Printpack has double-sided migration studies of the pouch materials for MWS in last year's chicken and dumplings and this year's Salmon & Alfredo sauce using the US FDA "Chemistry Guidelines" for its "Food Contact Material Notification" program (see ANNEX B).

   The WSU MWS processes for mashed potatoes and salmon & Alfredo defined acceptable methods for inoculating the product with appropriately thermal resistant- spores cold spot of the tray and pouched packages. Identifying the cold spot in a pouch of heterogeneous components is a similar process, each of the elements of the diverse chicken and dumpling food product has its distinct thermal heating properties. MWS process validation effort has identified the dielectric properties of its component foods. Next steps involved determining the package cold spot using a model food and developing the techniques for heat resistant spore inoculation.

   Risk mitigation: WSU personnel replaced two unreliable microwave generators on its pilot line and recalibrated the modified line to the original one in October 2010. They completed cold spot and progressed to extended run trials as well as inoculated package studies. Progress report is presented in Annex A.

5. Tasks 8,9 (Shelf life Modeling): (None… task complete)
6. Tasks 10,11 (TTI Technology)

   TASK 10 (Immediate Solutions): (None… task complete)

   TASK 11 (Intermediate Solutions)

   The time temperature indicator system used to print an indicator message on packaging material must resist not only premature development of indicia during package material conversion, material shipment and storage, product packaging and sealing but also early message fade during post-processing shipment and storage. The formulation with its TTI functionality must be acceptable in Printpack's printing presses. Attempts to mimic full-scale press operations with a combination of printing on a press and

hand printing experienced some set-backs due to lack of clarity in the required order of inks.

Risk mitigation: Printpack and Segan completed the evaluation of three Segan chemistries in label form in WSU pilot plant runs. Segan prepared selected chemistries in Printpack's thermally-resistant ink vehicle and Segan developed a bench-top lamination simulation to anticipate their printing and laminating compatibility of this formulation must be acceptable in a commercial printing process. Confusion over the printing order of inks required for a functioning TTI was clarified and material has been successfully printed. Pouching and processing has been completed and evaluation is taking place.

Program Risks

1. Task 1 (Laminations): (None…this task is complete)
2. Task 2 (Physical, Barrier, Optical Data): (None…this task is complete)
3. Task 3 (Photodegradation Data): (None…this task is complete)
4. Tasks 4,5,6,7 (Packaged Products): WSU has recently been informed that chicken & dumplings entrée requires USDA approval rather than FDA approval. Final approval could be delayed until USDA has undergone training to familiarize the required staff with the technology. Progress report is presented in Annex A.
5. Tasks 8 & 9 (Shelf life Modeling): (None…this task is complete)
6. Tasks 10 &11 (TTI Technology):

   TASK 10 (Immediate Solutions): (None… task complete)

   TASK 11 (Intermediate Solutions): With the indicated mitigation plan for the technical risks for these tasks, we recognize little to no program risk for them.

Issues

Unexpected Issues

TASK AREA	COMMENT
1. Task 1 (Laminations):	(none)
2. Task 2 (Barrier data):	(none)
3. Task 3 (Photodegradation Data)	(none)
4. Tasks 4,6,7 (MWS process)*	• Qualitative study of extractables from unprocessed, MWS processed, and re-torted pouches indicates no significant concerns, but has lead to refinement of future protocols for US FDA food contact material compliance. (Annex A) • WSU has recently been informed that chicken & dumplings entrée requires USDA approval rather than FDA approval. Final approval could be delayed until USDA has undergone training to familiarize the required staff with the technology. The report on the filing should be delivered on time. • The main contact at WSU, Galina Mikhaylenko, has resigned her position. A new hire has been made to take over her activities with contracts and the consortium.
5. Tasks 8,9 (Shelf life Modeling)	(none)
6. Tasks 10,11 (TTI Technology)	(none)

Details

Good News

TASK AREA	COMMENT
1. Task 1 (Laminations):	• *New improved WVTR films from Toppan (GL-ARHF) provided expected improved results.** • *OTR performance at or below NSRDEC targets from last year duplicated this year**
2. Task 2 (Barrier data): 3. Task 3 (Photodegradation Data)	• *Photodegradation data indicates that 2 layers of pigmented adhesive is sufficient to protect lipids from photooxidation**
4. Tasks 4,6,7 (MWS process)	• *The WSU MWS pilot line will be available for trials throughout all of 2010.** • *Polymeric laminations for food processed produced with improved visual quality and full functionality.** • *Pouches and roll stock for Salmon & Alfredo sauce successfully used for validation trials**. • Chicken and dumpling trials are nearing completion and progress report is presented in Annex A.
5. Tasks 8&9 (Shelflife modeling)	• *Significant progress made in calibrating laboratory data for raw materials and polymeric lamination and gathering characteristic food data** • *Foil laminate structure has successfully been approximated in the system**
6. Task 10 (Immediate TTI Technology)	• *Work essentially complete and successful**
7. Task 11 (Intermediate TTI Technology)	• *Bench top testing of TTI pigment with standard flexographic heat-resistant vehicle confirmed functionality equal to or better than in previous screen printing vehicle** • Bench top laminations successfully processed through WSU MWS process.

* *Previously reported*

Details

Technical

TASK AREA	COMMENT
1. Task 1 (Laminations):	▪ Laminations complete (previously submitted).
2. Task 2 (Barrier data):	▪ Barrier data has been accepted previously.
3. Task 3 (Photodegradation Data)	▪ Photodegradation Data complete (previously submitted). ▪ Other physical data is complete for the 10 laminations (submitted with ANNEX A). ▪ WSU has completed its thin film MW resonance testing on the 10 laminations (previously submitted)
4. Tasks 4,6,7 (MWS process)	▪ The WSU MWS process has received full US FDA validation for a mashed potato product packaged in a plastic barrier tray and transparent lidding and for Salmon & Alfredo using Printpack pouches. ▪ Printpack has successfully laminated all polymeric high barrier structures for both shelf-stable thermally-processed food and hot fill items. ▪ WSU has processed all the pouches necessary for the deliverables and has begun inoculated package studies. Progress report is presented in Annex A.
5. Tasks 8,9 (Shelf life Modeling)	▪ Barrier performance for laminations and raw material base films have been validated in model by model developer. Full structures have been developed and analyzed using the model system.
8. Task 10 (Immediate TTI Technology)	▪ Final report with successful results previously submitted.
9. Tasks 11 (Intermediate TTI Technology)	▪ In depth evaluations of precise and repeated color indications finished on bench top and onto the WSU Pilot Plant for additional confirmation. ▪ Press run and pilot plant lamination is complete. Pouches have been processed at WSU and are currently undergoing evaluation at Printpack.

Financial

Project Expenses as of 31 March 2011

COST ELEMENT	Contract	Q-1 Amt	Q-2 Amt	Q-3 Amt	Q-4 Amt	Q-5 Amt	Q-6 Amt	Q-7 Amt	to Date
otal Direct Labor	232,930	28,496	24,112	34,396	6,635	7,438	6,672	1,990	109,739
ayroll Tax/Benefits	84,511	10,259	8,680	12,383	2,389	2,678	2,402	716	39,506
ept Overhead	135,996	16,527	13,984	19,950	3,849	4,314	3,870	1,155	63,649
Labor Total	453,437	55,282	46,776	66,729	12,873	14,430	12,944	3,861	212,894
onsulting/Services	544,500	0	941	87,909	68,441	217,532	112,750		486,632
ltls/Plant Costs	70,850	33,170	-	16,153	158,940	0	34,212	0	242,475
ravel	24,530	6,660	4,152	5,188	1,980	900	828	3,035	22,743
ther Direct Costs	160,760	39,830	5,093	109,250	229,361	218,432	147,790		751,850
Total Costs	1,254,077	95,112	51,869	175,979	242,234	232,265	160,734		964,744
10% Fee	125,409	9,511	5,187	17,598	24,223	23,227	16,073		96,474
Contract Total	1,379,486	104,623	57,056	193,577	266,457	255,492	176,807		1,061,218

Project expenses to date as captured in Printpack's financial accounting system indicate that the work remains on budget. Some of the budgeted direct labor costs are reported here as plant costs. Most of the remaining work will be conducted by the subcontractors.

Equipment*

(No change from last report)

Equipment	**Cost**	**Assoc. Task(s)**	**Location**
OXTRAN® 2/21 SL Env. Chamber Operating. System	$61,641	2	Printpack Analytical Services Lab 5 Barber Industrial Ct. Villa Rica, GA 30180
Leap Autosampler System	$36,107.	3	

* No additional equipment purchases during quarters subsequent to 1st.

SUBCONTRACTS

Subcontracts

Subcontractor	Cost	Assoc. Task(s)	Status
Washington State University	$400,533	4,6,7	Work underway; no-cost time extension negotiated with WSU and proposed to DOD
Segan Industries	$144,000.	10,11	Phase I and Phase II complete for deliverables. Ongoing research to optimize color reference chart for MWS temperatures.

Amended timing for WSU deliverables as negotiated with Printpack and proposed to DOD is…

Deliverables	Delivery Date
1. Report on thermal stability of selected TTI label materials in high temperature environment suitable for thermal sterilization applications. We may use WSU package film test cells and oil baths to conduct heating tests. Samples will be evaluated at WSU and in Printpack. (See ANNEX C)	Oct 31,2010
2. Report on interaction among microwaves, food package films and foods; with evaluation of thermal stability of Printpack films using 40 kW 915 MHz microwave sterilization system at WSU. Send processed films to Printpack for quality evaluation. Formulation of the standard test food model will be developed in cooperation with Printpack. (See ANNEX D)	Oct 31,2010
3. Developed and validated thermal processing procedures for two food products (chicken dumpling and salmon in Alfredo sauce) in three selected film materials using the 915 MHz microwave sterilization system.	June 30,2011
4. Produce food products in pouches, conduct microbial check, and send 40 pouches of each product to Natick for sensory and shelf-life studies. We will also process the same foods in MRE foil pouches using conventional retorting method for comparison.	Mar 31,2011
5. Assistance to Printpack in studies of commercial scale-up abilities.	Jul 31,2011
6. Assistance to Printpack in testing TTI system for package materials compatible with microwave sterilization system.	Nov 30,2010

ANNEX A

Deliverables 4, 6, 7: Chicken and Dumplings Entrée Report

WASHINGTON STATE UNIVERSITY
World Class. Face to Face.

REPORT ON PROGRESS OF CHICKEN-DUMPLING-POUCH PROJECT

Submitted to PrintPack

By

WSU MW Group

Department of Biological Systems Engineering
Washington State University
June, 2011

This report summarizes major progress of the project "Microwave Sterilization of Chicken and Dumplings packaged in 8-oz Pouches" for the period between January and June, 2011.

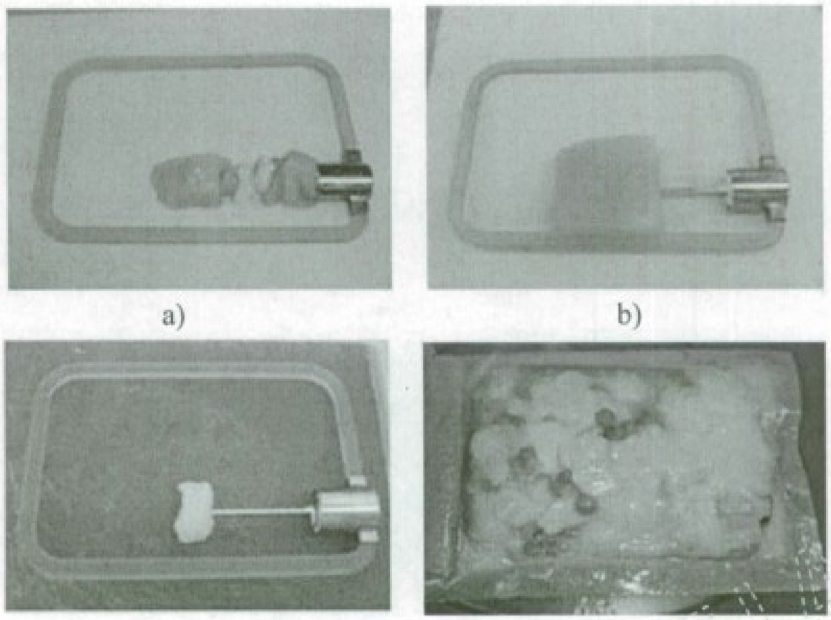
1. Identification of the Food Component Getting the Lowest Thermal Lethality

The chicken – dumpling pouch we developed is filled with four components: cut chicken breast pieces, dumplings, sauce, and vegetables. The components have different dielectric properties, thermal properties and heat resistances to microorganisms. In heat penetration tests, the temperature sensor needs to be placed in the component located at the cold spot which obtains the lowest thermal lethality, to monitor the food temperature. Therefore, it is necessary to identify the food component receiving the lowest thermal lethality.

MW processing tests for measuring temperatures inside the three different components (chicken breast pieces, dumplings, and sauce) placed at the cold spot (identified by the chemical-marker-based computer-vision method) inside pouches were conducted. The pouches were filled with designated rations of the following food components: 95 g chicken breast, 80 g sauce, 16 g dumplings, and 7 g vegetables. Ellab sensors were used to measure the temperatures profiles (Figs. 1.1 & 1.2). Two sizes of cut chicken pieces were used for the temperature measurement: a normal size of 16×16×16 mm and a larger size of 40×40×16 mm.

Tests were conducted under the following conditions:

- 8-oz PrintPack pouches
- MW power setting: 7.0 / 6.2 / 2.6 / 2.5 kW for 4 MW heating cavities
- Moving speed: 40 inch/min
- Water temperature: 72 / 124 / 123°C for preheating, MW heating and holding sections
- System pressure: 34 psig
- Water flow rate: 69 / 51 / 72 / 61 liter/min for pre-heating, MW heating, holding, and cooling sections
- Pre-heating time: 30 min
- Cooling time: 4 min.



a) b) c) d)

Fig. 1.1 Placement of Ellab sensor. a) sensor tip inside chicken piece of normal size (16×16×16 mm) at cold spot; b) sensor tip inside chicken piece of larger size (40×40×16 mm) at cold spot; c) sensor tip inside dumpling at cold spot; d) sample pouch with Ellab sensor after processing.

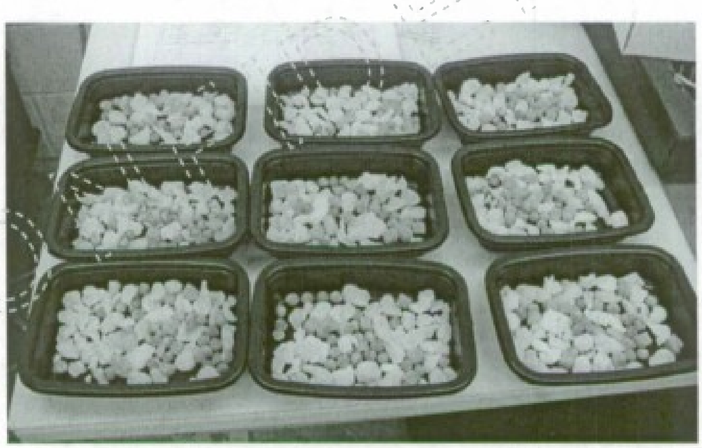


Fig. 1.2 Dumpling and vegetables

F_0 values achieved in different components at the cold spot location were determined based on the measured temperature profiles (Tables 1.1 and 1.2). Results show that chicken pieces receive less thermal treatment at the cold spot than dumplings or sauce. In addition, chicken has higher heat resistance to microorganisms than dumplings or sauce as mentioned in Section 4.1. Both lower thermal treatment and higher heat resistance to microorganisms cause chicken pieces to receive lower thermal lethality. Therefore, chicken pieces were chosen as the target component for temperature measurement in MW processing development.

Table 1.1 F_0 values in different components at cold spot in pouches with a normal-size chicken piece for temperature measurement

date	F0, min chicken	F0, min dumpling	F0, min sauce
Test-1, Jan-12-2011	11.4	20.2	19.8
	15.9		
Test-2, Jan-12-2011	20.8	15	23.9
	22.2		
Test-3, Jan-12-2011	13.6	19.6	21.3
	13.6		
average F0, min	16.3	**18.3**	**21.7**
Stdev	**4.3**	**2.8**	**2.1**

Table 1.2 F_0 values in different components at cold spot in pouches with a larger-size chicken piece for temperature measurement

testing date	F0, min chicken	F0, min dumpling	F0, min sauce
Test-2, Jan-11	6.2	25	20.8
	9.5		
Test-3, Jan. 11	8.1	26.2	23.7
		24.1	
Test-1, Jan. 14	6.5		
	7.7	15.5	33.9
average F0, min	7.6	**22.7**	**26.1**
Stdev, min	**1.3**	**4.9**	**6.9**

2. Validation of Cold Spot by Temperature Measurement inside Chicken Dumpling Pouches

2.1. Temperature measurement at cold regions in chicken dumpling pouches

Tests were conducted to measure temperature profiles and F_0 values inside chicken pieces (40×40×16 mm) in chicken dumpling pouches using Ellab sensors. The chicken pieces with Ellab sensors were placed in three cold regions identified by the chemical-marker-based computer-vision method. The tips of the Ellab sensors were placed at central points (P2, P3, and P6) of the cold regions (Figs. 2.1 & 2.2).

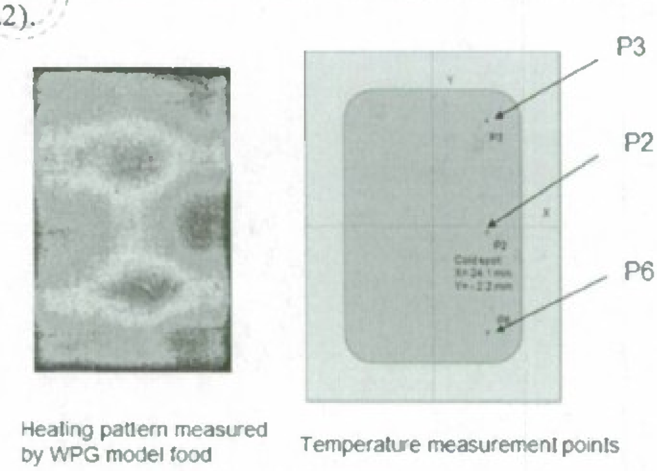


Fig. 2.1 Measuring points in cold regions

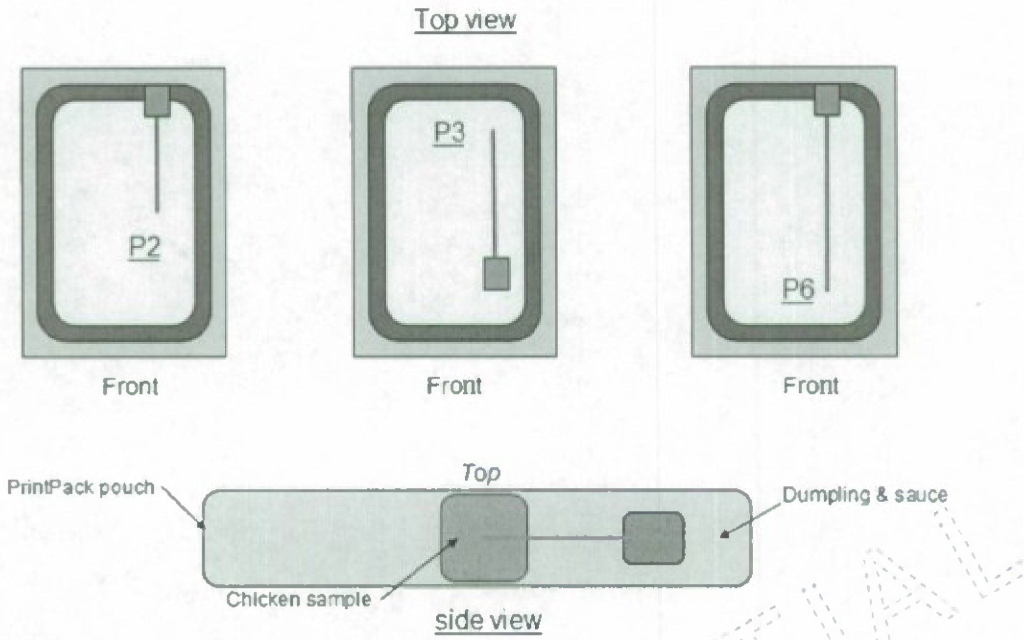


Fig. 2.2 Measuring points with Ellah sensors

Tests were conducted under the same conditions described in Section 1. Table 2.1 shows the measured F_0 values. The F_0 at point 2 (identified cold spot by chemical-marker-based computer-vision method for WPG samples) in the chicken-dumpling pouch had the lowest value.

Table 2.1 F_0 values measured at 3 points in 3 cold regions in chicken-dumpling pouches

Testing date	F0, min at P2	F0, min at P3	F0, min at P6
Test-3, Jan. 12, 2011	9.1		13.6
	6.2	21.3	
Test-1, Jan. 13, 2011	6.9	17.9	18.3
		11.8	
Test-2, Jan. 13, 2011	7.3	19.2	12.7
			12.6
average F0, min	7.4	**17.6**	**14.3**
Stdev, min	**1.2**	**4.1**	**2.7**

2.2. Temperature measurement at points surrounding the identified cold spot in chicken-dumpling pouches

To further confirm the cold spot identified by the chemical-marker-based computer-vision method for WPG samples to be the actual cold spot inside chicken-dumpling pouches, temperature profiles

and F_0 values at the identified cold spot (Point 2) and its surrounding points (up, down, front, back, left, and right, 4 or 5 mm away from the cold spot) were measured using Ellab sensors. Figure 2.3 shows the locations of the measuring points inside the chicken piece (40×40×16 mm) in chicken-dumpling pouches.

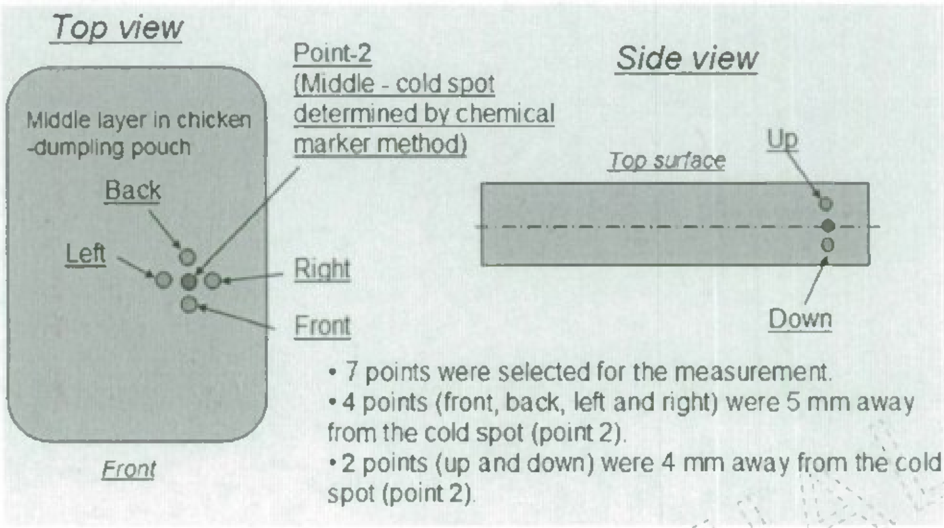


Fig. 2.3 Location of measuring points

Testing conditions were same as those stated in Section 1. Testing results (Table 2.2) indicate that MW processing provided higher thermal treatment at the surrounding points than at Point 2. The cold spot identified by the chemical-marker-based computer-vision method is indeed the actual cold spot inside the chicken-dumpling pouches.

Table 2.2 F_0 values at identified cold spot and its surrounding points

Location	P2	Up	Down	Front	Back	Left	Right
F0, min	9.11	8.81	9.37	14.19	15.04	7.67	7.1
	6.22	9.22	7.72	9.42	14.46	8.6	9.8
	6.2	6.94	8.64	10.98	14.05	16.61	18.96
	7.34	13.12	7.09	20.38	23.9	24.8	14.79
	6.51		10.03				
	7.71						
	6.81						
	8.21						
	10.15						
Average	**7.58**	**9.52**	**8.57**	**13.74**	**16.86**	**14.42**	**12.66**
Stdev	**1.29**	**2.25**	**1.07**	**4.20**	**4.08**	**6.93**	**4.56**

3. Heat Penetration Tests

Heat penetration (HP) tests were conducted to achieve a target F_0 of 6.0 minutes inside the chicken piece (40×40×16 mm) placed at the cold spot in chicken-dumpling pouches at the end of complete thermal process. The 8-oz PrintPack pouch was filled with food components of the pre-selected ration (95 g chicken breast pieces, 80 g sauce, 16 g dumplings, and 7 g vegetables) and sealed with an UltraVac 250 vacuum pouch sealer (KOCH Packaging Supplies Inc., Kansas City, MO) under pre-selected conditions (vacuum setting: 2.5; sealing time setting: 4). The size of cut chicken pieces was 16×16×16 mm except for the piece for temperature measurement at the cold spot. Temperature profiles in the chicken piece at cold spot were measured by Ellab sensors during the tests. The test parameters and conditions were same as those stated in Section 1.

A total of 37 data points were collected from 19 HP tests runs conducted over 17 days. Figure 3.1 shows sample temperature profiles measured by Ellab sensors during one test. Table 3.1 summarizes F_0 values obtained from all the tests. The F_0 varied from 6.2 to 15.1 min during the tests performed with the selected processing schedule. The thermal contribution during the cooling period was considered in the F_0 calculation for the HP tests. Table 3.2 summarizes the important processing parameters for the 37 data sets including MW power, processing time, and water temperature in heating and holding sections.

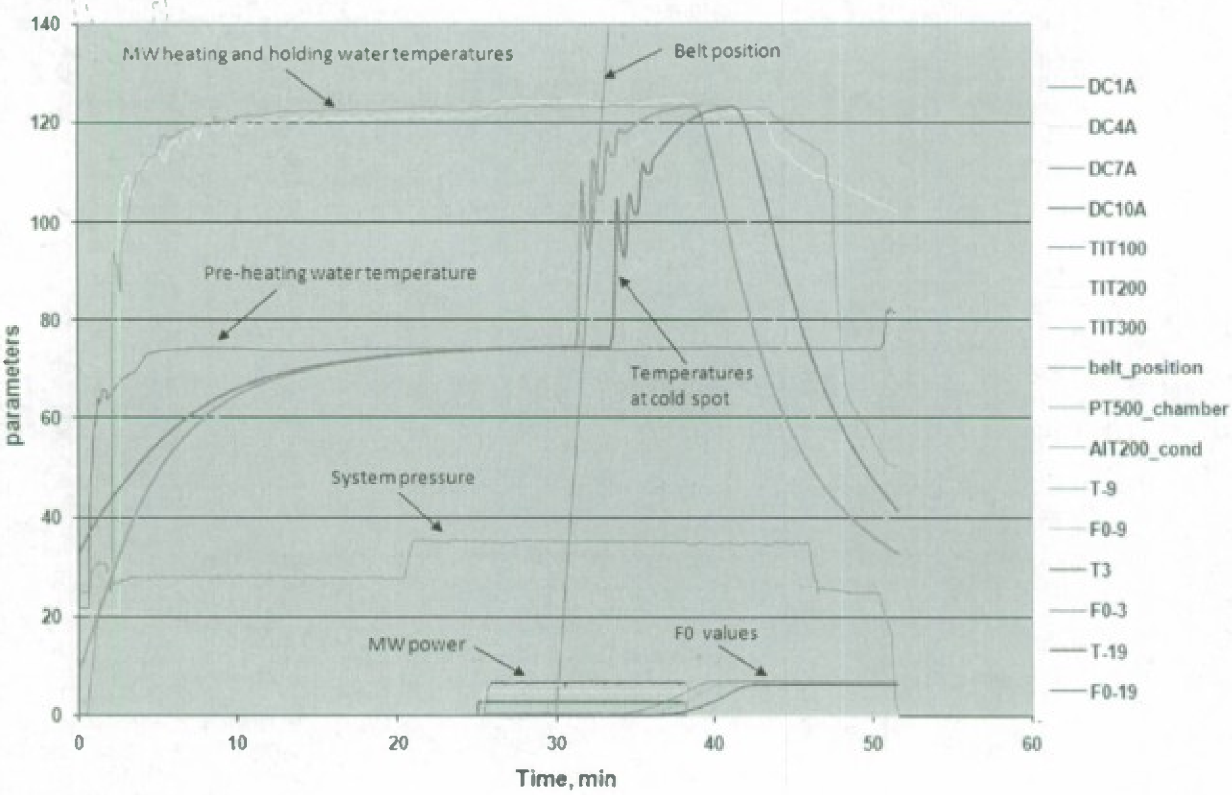


Fig. 3.1 Sample temperature profiles measured by four Ellab sensors during one test.

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Table 3.1 Location of pouches on the mesh belt and F_0 at the cold spot of the pouches at the end of processing

Testing date	Jan 12-11 test3	Jan 13-11 test1	Jan 13-11 test2	Jan 14-11 test1	Jan 14-11 test2	Jan 18-11 test2	Jan 18-11 test3	Jan 19-11 test1	Jan 21-11 test1	Jan 24-11 test1	Jan 24-11 test2	Jan 25-11 test1	Jan 25-11 test2	Jan 26-11 test1	Jan 26-11 test2	Jan 27-11 test1	Jan 27-11 test2	Jan 28-11 test1	Jan 28-11 test2
Pouch location on the mesh belt	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min	F0, min
1																			
2																			
3																8.86			
4																			
5																			12.65
6	9.11			6.51	6.81														
7															6.18				
8														7.65					
9																8.89			
10			7.34																
11														8.21					
12	6.22	6.9		7.71															
13															8.95				
14																	15.14		
15											12.23								
16														7.92					
17															12.19				
18						8.21												14.38	
19																6.28			
20																			7.15
21																			
22							10.15		7.58										
23																	11.23		
24								13.56											
25										8.81									
26																		14.06	
27									6.45										
28																			
29													9.01						
30																			
31																			
32											14.71								
33																		13.36	
34													9.14						
35																	14.00		
36																			
37										9.86									
38																			
39																			13.65
40												14.44							
41																			
42																			

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Table 3.2 MW HP Test Matrix and Data Summary

Microwave Assisted Sterilizer - Heat Penetration Test Data Summary

MW Section Lengths
Preheat: 20.0 ft
MW Heating: 10.625 ft
Holding: 10.917 ft

Belt Speed: 3.33 ft/min

	Run / TC	I.T. (°F)	Preheat Time (min)	Max.* Preheat Temp. (°F)	Time in Heating Section (min)	Max.* Temp. in Heating Section (°F)	Time in Holding Section (min)	Max.* Temp. in Holding Section (°F)	Max.* MW Power Cavity 1 (kW)	Max.* MW Power Cavity 2 (kW)	Max.* MW Power Cavity 3 (kW)	Max.* MW Power Cavity 4 (kW)	General Method Lethality (F_0) *	Belt Speed (ft/min)	System Pressure (psig)	Fill Wt of Chicken (oz)	Fill Wt of Dumpling (oz)	Fill Wt of Vegetable (oz)	Fill Wt of Sauce (oz)
1	Jan/12/11-T3/6	43.97	30.00	164.61	3.19	256.17	3.26	254.93	7.16	6.32	2.59	2.55	9.11	3.35	34.85	3.35	1.27	0.56	2.82
2	Jan/12/11-T3/12	44.31	30.00	164.61	3.19	255.79	3.28	255.00	7.18	6.32	2.59	2.55	6.22	3.35	34.85	3.35	1.27	0.56	2.82
3	Jan/13/11-T1/12	43.16	30.00	165.00	3.19	256.08	3.26	255.09	7.26	6.44	2.59	2.53	6.90	3.35	34.92	3.35	1.27	0.56	2.82
4	Jan/13/11-T2/10	42.75	30.00	164.62	3.19	256.41	3.26	255.06	7.22	6.42	2.60	2.54	7.34	3.35	34.96	3.35	1.27	0.56	2.82
5	Jan/14/11-T1/06	41.76	30.00	165.02	3.19	256.59	3.26	254.70	7.28	6.44	2.59	2.59	8.51	3.35	34.96	3.35	1.27	0.56	2.82
6	Jan/14/11-T1/12	41.29	30.00	165.02	3.19	256.44	3.26	254.70	7.26	6.44	2.59	2.59	7.71	3.35	34.96	3.35	1.27	0.56	2.82
7	Jan/14/11-T2/06	43.25	30.00	165.02	3.19	256.26	3.26	254.70	7.17	6.38	2.59	2.59	6.81	3.35	34.86	3.35	1.27	0.56	2.82
8	Jan/18/11-T2/16	44.69	30.00	165.06	3.19	256.64	3.26	254.80	7.28	6.42	2.59	2.54	8.21	3.35	34.75	3.35	1.27	0.56	2.82
9	Jan/18/11-T3/22	43.56	30.00	165.00	3.19	255.87	3.26	254.46	7.18	6.39	2.59	2.56	10.15	3.35	34.72	3.35	1.27	0.56	2.82
10	Jan/19/11-T1/24	40.51	30.00	165.27	3.19	255.72	3.26	254.60	7.20	6.35	2.59	2.55	13.56	3.35	35.25	3.35	1.27	0.56	2.82
11	Jan/21/11-T1/22	39.56	30.00	165.15	3.19	256.19	3.26	254.46	7.24	6.40	2.59	2.62	7.56	3.35	34.84	3.35	1.27	0.56	2.82
12	Jan/21/11-T1/27	39.89	30.00	165.15	3.19	256.01	3.26	254.57	7.24	6.40	2.59	2.62	6.45	3.35	34.64	3.35	1.27	0.56	2.82
13	Jan/24/11-T1/25	38.93	30.00	165.16	3.19	256.69	3.26	254.62	7.21	6.40	2.59	2.56	6.61	3.35	34.67	3.35	1.27	0.56	2.82
14	Jan/24/11-T1/37	39.47	30.00	165.16	3.19	256.46	3.26	255.09	7.21	6.40	2.59	2.56	9.88	3.35	34.67	3.35	1.27	0.56	2.82
15	Jan/24/11-T2/15	40.06	30.00	165.02	3.19	255.90	3.26	254.71	7.25	6.39	2.59	2.57	12.23	3.35	34.77	3.35	1.27	0.56	2.82
16	Jan/24/11-T2/32	40.08	30.00	165.02	3.19	256.62	3.26	254.93	7.25	6.39	2.59	2.57	14.71	3.35	34.77	3.35	1.27	0.56	2.82
17	Jan/25/11-T1/40	40.12	30.00	166.05	3.19	256.77	3.26	255.11	7.27	6.39	2.59	2.57	14.44	3.35	34.62	3.35	1.27	0.56	2.82
18	Jan/25/11-T2/29	41.07	30.00	165.54	3.19	256.80	3.26	254.98	7.17	6.38	2.59	2.63	9.01	3.35	34.79	3.35	1.27	0.56	2.82
19	Jan/25/11-T2/34	41.38	30.00	165.54	3.19	257.09	3.28	254.98	7.17	6.36	2.59	2.63	9.14	3.35	34.79	3.35	1.27	0.56	2.82
20	Jan/26/11-T1/08	40.64	30.00	165.74	3.19	256.19	3.26	254.61	7.29	6.42	2.59	2.56	7.65	3.35	34.93	3.35	1.27	0.56	2.82
21	Jan/26/11-T1/11	40.64	30.00	165.74	3.19	255.72	3.28	254.61	7.29	6.42	2.59	2.56	8.21	3.33	34.93	3.35	1.27	0.56	2.82
22	Jan/26/11-T1/16	40.76	30.00	165.74	3.19	256.19	3.26	254.61	7.29	6.42	2.59	2.56	7.92	3.33	34.93	3.35	1.27	0.56	2.82
23	Jan/26/11-T2/07	41.22	30.00	165.69	3.19	256.42	3.26	254.75	7.22	6.42	2.59	2.56	6.16	3.35	34.79	3.35	1.27	0.56	2.82
24	Jan/26/11-T2/13	42.46	30.00	165.69	3.19	256.01	3.26	254.97	7.22	6.42	2.59	2.56	8.95	3.35	34.79	3.35	1.27	0.56	2.82
25	Jan/26/11-T2/17	41.47	30.00	165.69	3.19	256.86	3.26	254.97	7.22	6.42	2.59	2.56	12.19	3.33	34.79	3.35	1.27	0.56	2.82
26	Jan/27/11-T1/03	41.18	30.00	165.70	3.19	256.41	3.28	254.37	7.16	6.35	2.59	2.57	6.88	3.35	35.00	3.35	1.27	0.56	2.82
27	Jan/27/11-T1/09	41.13	30.00	165.70	3.19	255.81	3.26	254.75	7.18	6.35	2.59	2.57	6.89	3.35	35.00	3.35	1.27	0.56	2.82
28	Jan/27/11-T1/19	42.03	30.00	165.70	3.19	256.35	3.26	254.84	7.18	6.35	2.59	2.57	6.26	3.35	35.00	3.35	1.27	0.56	2.82
29	Jan/27/11-T2/14	40.59	30.00	165.45	3.19	255.88	3.26	254.61	7.20	6.37	2.59	2.56	15.14	3.33	34.65	3.35	1.27	0.56	2.82
30	Jan/27/11-T2/23	39.97	30.00	165.45	3.19	256.06	3.26	254.61	7.20	6.37	2.59	2.56	11.23	3.33	34.65	3.35	1.27	0.56	2.82
31	Jan/27/11-T2/35	40.64	30.00	165.45	3.19	256.59	3.26	255.16	7.20	6.37	2.59	2.56	14.00	3.33	34.65	3.35	1.27	0.56	2.82
32	Jan/28/11-T1/16	38.57	30.00	165.69	3.19	255.51	3.26	254.35	7.16	6.36	2.59	2.56	14.38	3.35	34.67	3.35	1.27	0.56	2.82
33	Jan/28/11-T1/26	38.66	30.00	165.69	3.19	255.70	3.26	254.56	7.16	6.36	2.59	2.56	14.06	3.35	34.67	3.35	1.27	0.56	2.82
34	Jan/28/11-T1/35	38.41	30.00	165.69	3.19	256.06	3.26	255.18	7.16	6.36	2.59	2.56	13.38	3.35	34.67	3.35	1.27	0.56	2.82
35	Jan/28/11-T2/05	41.07	30.00	165.70	3.19	256.64	3.26	254.35	7.24	6.38	2.59	2.59	12.65	3.35	34.69	3.35	1.27	0.56	2.82
36	Jan/28/11-T2/20	41.52	30.00	165.70	3.19	256.01	3.29	254.53	7.24	6.38	2.59	2.59	7.15	3.35	34.69	3.35	1.27	0.56	2.82
37	Jan/28/11-T2/39	41.07	30.00	165.70	3.19	256.73	3.26	255.00	7.24	6.38	2.59	2.59	13.65	3.35	34.89	3.35	1.27	0.56	2.82
	HP Values	44.69	30.00	166.05	3.19	257.09	3.26	255.18	7.29	6.44	2.60	2.63	8.16	3.35	35.25	8oz			

4. Microbiological Validation of MW Process

4.1. Update of microbial work – determination of D- and z-values of PA 3679 *Clostridium sporogenes* spores in chicken breast

Raw skinless chicken breast was ground in a small electric food blender for 2 min. Ten g of blended chicken was placed in a 50 ml disposable conical centrifuge tube. Five hundred µl of PA 3679 # 308 *Clostridium sporogenes* spore crop (Mah July 2007 bottle no. 1) was placed in a depression formed in the chicken. A sterile metal spatula was used to thoroughly mix the spores and chicken for 10 min. A hypodermic syringe with a snipped yellow 0-200 µl pipette tip was used to inject chicken to a length of 50 mm inside a 1.8 mm glass capillary tube, then a 1.5 mm glass capillary tube was used to transfer the sample to the center of the 1.8 mm capillary tube. A lightly alcohol-wetted piece of Kimwipe wrapped around a 24 ga steel wire was used to clean traces of chicken from the end of the tube, to facilitate better flame-sealing with a Bunsen burner. Heat

treatment was performed at pre-selected temperatures (113.0, 115.0, 118.0, and 121.1°C) in an oil bath for a variety of time intervals appropriate for each selected temperature. A 12 sec come-up time was included for all temperatures studied. Samples were cooled immediately for 2 min in an ice-water bath. Capillary tubes were opened aseptically with a file and contents were transferred to pre-weighed 15 cm conical centrifuge tubes containing 3 ml of sterile 0.1% peptone water. Tubes containing chicken were re-weighed and net weight of chicken was calculated. Chicken was homogenized by hand using the base of a sterile metal transfer loop handle or flame-polished glass rod using a grinding motion against the centrifuge tube bottom. Tenfold serial dilutions were performed using tubes containing 4.5 ml of 0.1% sterile peptone water. One ml of appropriate dilutions was duplicate spread-plated with TPGY agar and incubated 3 days at 32°C under anaerobic conditions. Plate counts were taken and CFU/ml and CFU/g chicken were calculated. Experiments were replicated 2-3 times.

Average D-values for the different temperatures were calculated and the z-value determined (Table 4.1 & Fig. 4.1). The D-value of PA 3679 spores in chicken breast at 121.1°C was 0.97 min; the z-value of PA 3679 spores in chicken breast was determined to be 9.26°C.

D-values of PA 3679 spores in dumplings and sauce at 121.1°C were determined previously: 0.47 min and 0.68 min, respectively.

Table 4.1 Summary of D-value results

Temp, °C	D-value, min					Log_{10} D-value				
	Rep 1	Rep 2	Rep 3	Average	Std Dev	Rep 1	Rep 2	Rep 3	Average	Std Dev
121.1	0.95	0.99		0.97	0.03	-0.02	0.00		-0.01	0.01
118	2.99	2.30	2.03	2.44	0.50	0.48	0.36	0.31	0.38	0.09
115	5.01	5.03		5.02	0.01	0.70	0.70		0.70	0.00
113	5.89	7.95	7.66	7.17	1.12	0.77	0.90	0.88	0.85	0.07

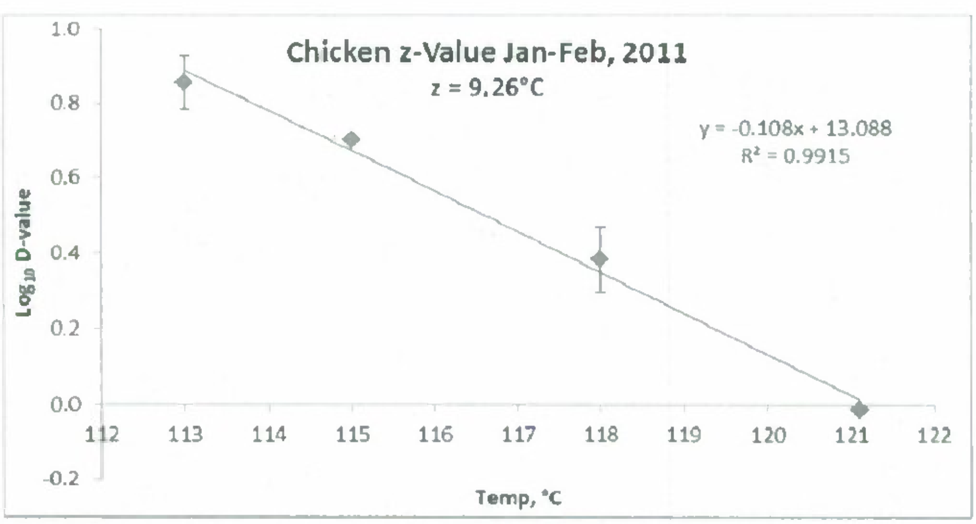

Fig. 4.1 Test results for determination of z-value of PA 3679 spores in chicken breast

4.2. Development of processing schedules for inoculated pack studies

Systematic tests were conducted with different belt speeds to achieve different F_0 values at the cold spot inside chicken-dumpling pouches. After each test run, F_0 values for the selected pouches were determined by the general method based on the temperature profiles measured by the Ellab sensors. The thermal contribution during the cooling period was included in the calculation of the F_0 values. The moving speeds of the food pouches were selected for achieving the F_0 of 2.4, 4.2, 6.2, and 8.6 min. Table 4.2 summarizes the developed processing schedules.

Table 4.2 Processing schedules for inoculated studies

Process level	F_0, min	Moving speed, inch/min	MW power setting, kW			
			Cavity 1	Cavity 2	Cavity 3	Cavity 4
Level-1	2.4	46	7.0	6.2	2.6	2.5
Level-2	4.2	43	7.0	6.2	2.6	2.5
Level-3	6.2	40	7.0	6.2	2.6	2.5
Level-4	8.6	39	7.0	6.2	2.6	2.5

4.3. Selection of inoculation level for inoculated pack studies

Inoculation level is a critical parameter for inoculated pack studies. The inoculation level should be selected based on the following rule: there are surviving spores in all or most of the inoculated packages after processing under the lowest process level (Level 1); there are surviving spores in some of the inoculated packages after processing under a lower process level (Level 2); there are no surviving spores in any of the inoculated packages after processing under a higher process level (Level 3) and the highest process level (Level 4). An inoculation level of 5×10^3 CFU/pouch was selected for the inoculated pack studies in this project. Theoretically, after processing under process level 1, 2, 3, or 4, the number of spores surviving in each inoculated pouch are 17, 0.234, 0.002, and 0.0000068. Consequently, the chance of a single spore surviving in each inoculated pouch is 100%, 23.4%, 0.2% and 0.00068%, respectively (Table 4.3).

Table 4.3 Chance of spore survival under different process levels (with inoculation of 5×10^3 CFU/pouch)

Processing level	Moving speed, inch/min	F_0	Log_{10} reduction	Inoculation level option : 5×10^3 CFU/pouch	
				Expected number of surviving spores per pouch	Expected chance of survival of a single spore in a pouch
Level-1	46	2.4	2.474	16.7781163	100%
Level-2	43	4.2	4.330	0.2339231	23.4%
Level-3	40	6.2	6.392	0.0020287	0.20%
Level-4	39	8.6	8.866	0.0000068	0.00068%

4.4. Preparation of samples and inoculation of the pouches

Each 8-oz Printpack pouch was filled with 95 g cut chicken breast pieces, 80 g sauce, 16 g dumplings, and 7 g vegetables. Among the fillings, a piece of cut chicken breast (40×40×16 mm) was inoculated by pipetting 10 μl of spore crop containing 5×10^3 CFU spores and was placed at the cold spot inside the pouch (Fig. 4.2). The pouches were sealed with an UltraVac 250 vacuum pouch sealer (KOCH Packaging Supplies Inc., Kansas City, MO) using a custom program (vacuum setting: 2.5; sealing time setting: 4) permitting a small amount of residual air (less than 3.5 cc) in the package. For each test run, two pouches with an Ellab sensor placed at the cold spot were prepared. The sealed pouches were kept in a cold room (4°C) prior to MW sterilization.

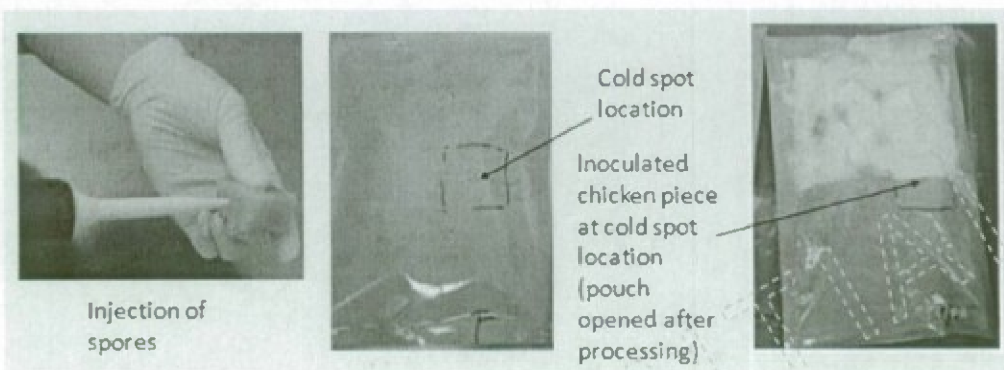

Fig. 4.2 Inoculation of sample and location of inoculated sample

4.5. Preliminary inoculated pack studies

Preliminary inoculated pack studies were conducted at three process levels (Level 1, 2, and 3) (Table 4.4). One test run was performed for each processing schedule. Five or ten inoculated pouches and two pouches with Ellab sensors were processed in each test. Five control pouches were inoculated with spores which were heat-shock activated by pre-treatment (heated for 20 min at 80°C then immediately cooled in ice-water). All the processed and un-processed control pouches were placed in a walk-in incubator at 36.5 ± 0.5°C for incubation (Fig. 4.3). Table 4.5 summarizes the observation results after 60 days of incubation (updated on April 30, 2011). All 5 control pouches swelled within 1 day due to gas production resulting from the growth of *C. sporogenes* PA 3679. Three out of 5 pouches processed at Level 1 (F_0 = 2.4 min) and 4 out of 10 pouches processed at Level 2 (F_0 = 4.2 min) swelled in 3 days. The other pouches have shown no evidence of gas production.

Table 4.4 Experimental design for preliminary inoculated pack studies

Processing level	Moving speed, inch/min	F_0	Inoculated pouches	Inoculum, CFU/pouch
Level-1	46	2.4	5	5×10^3
Level-2	43	4.2	10	5×10^3
Level-3	40	6.2	5	5×10^3
Control	un-processed		5	5×10^3

Fig. 4.3 MW processed and control pouches being incubated in walk-in incubator

Table 4.5 Incubation results for preliminary inoculated pack studies (updated on April 30, 2011; after 60 days' incubation)

Observation Sheet (preliminary chicken dumpling inoculated pack studies)													
Days of incubation		0	1	2	3	4	5	10	20	30	40	50	60
Observation date		1-Mar	2-Mar	3-Mar	4-Mar	5-Mar	6-Mer	11-Mer	21-Mer	31-Mer	10-Apr	20-Apr	30-Apr
	Pouch #1	Neg	B	B	B	B	B	B	B	B	B	B	B
Control with spores	Pouch #2	Neg	B	B	B	B	B	B	B	B	B	B	B
	Pouch #3	Neg	B	B	B	B	B	B	B	B	B	B	B
	Pouch #4	Neg	B	B	B	B	B	B	B	B	B	B	B
	Pouch #5	Neg	B	B	B	B	B	B	B	B	B	B	B
	Pouch #7	Neg	Neg	B	B	B	B	B	B	B	B	B	B
	Pouch #8	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
F_0= 2.4 (L1)	Pouch #9	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #11	Neg	Neg	B	B	B	B	B	B	B	B	B	B
	Pouch #12	Neg	Neg	B	B	B	B	B	B	B	B	B	B
	Pouch #7	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #8	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #9	Neg	Neg	B	B	B	B	B	B	B	B	B	B
	Pouch #10	Neg	Neg	Neg	B	B	B	B	B	B	B	B	B
F_0= 4.2 (L2)	Pouch #11	Neg	Neg	Neg	B	B	B	B	B	B	B	B	B
	Pouch #13	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #14	Neg	Neg	B	B	B	B	B	B	B	B	B	B
	Pouch #15	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #16	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #17	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #7	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #8	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
F_0= 6.2 (L3)	Pouch #9	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #11	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
	Pouch #12	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg	Neg
Observer(s):		FL, ZT, HL	FL, ZT, HL	FL, ZT, HL	FL, ZT, HL	HL	HL	HL	HL	HL	HL	HL	HL
Note: B-buiging; Neg-negative													
FL-Frank Liu; ZT-Zhongwei Tang; HL-Huimin Lin													

4.7. Final full-scale inoculated pack studies

Tests were conducted in two replicates under each of the four process levels (Table 4.6). In each test, 25 inoculated pouches, 5 un-inoculated pouches, 2 pouches with Ellab sensors, and 10 dummy pouches (5 placed at each end of the conveyor belt) were processed. A total of 8 runs of tests were performed for the inoculated pack studies. All the MW-processed and control pouches were moved into the walk-in incubator on March 25, 2011 for incubation (Fig. 4.4).

Table 4.6 Experimental design for full scale inoculated pack studies

Processing level	Moving speed, inch/min	F_0, min	Inoculated Pouches (per replicate)	Inoculum/ Pouch	Un-inoculated/ Treated Pouches (per replicate)	Replicate Runs	Total Pouches
Level-1	46	2.4	25	5×10^3	5	2	60
Level-2	43	4.2	25	5×10^3	5	2	60
Level-3	40	6.2	25	5×10^3	5	2	60
Level-4	39	8.6	25	5×10^3	5	2	60
Control	un-processed		30	5×10^3	N/A	N/A	30
Total							**270**

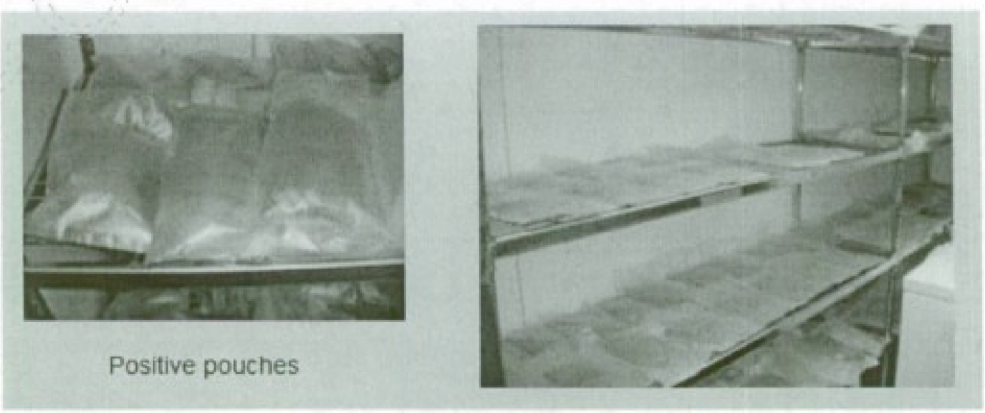

Fig. 4.4 MW processed and control pouches placed in walk-in incubator for incubation.

The walk-in incubator was controlled at 36.5 ± 0.5°C (Fig. 4.5), which was monitored every 5 days with 3 thermometers placed at different locations and recorded on a 24-h circular chart recorder. The pouches were / are / will be observed every day for the first 5 days and every 5 days thereafter for 3 months.

Table 4.7 summarizes the observation results after 80 days of incubation (updated on June 13, 2011). All 30 untreated inoculated control pouches swelled within 1 day due to gas production resulting from the growth of *C. sporogenes* PA 3679. All the 10 un-inoculated pouches processed at each level were negative. Bulging was detected in 27 of 50 inoculated pouches (54%) processed under Level 1 (F_0 = 2.4 min), and in 2 of 50 pouches (4 %) processed under Level 2 (F_0 = 4.2 min), respectively. The inoculated pouches processed under Level 3 & 4 (F_0 = 6.2 & 8.6 min) have shown

no evidence of gas production. The incubation results suggest that the MW sterilization processing delivered expected lethalities to *C. sporogenes* PA 3679 spores.

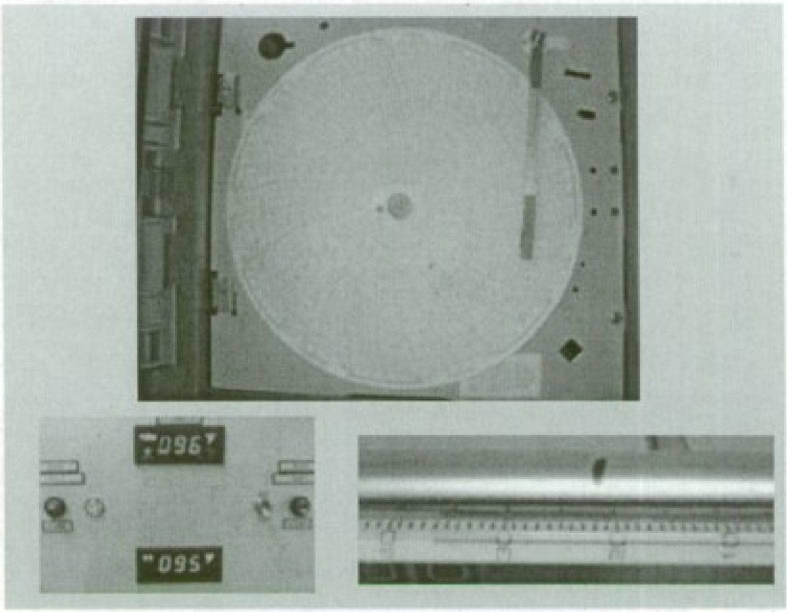

Fig. 4.5 Temperature controlled for incubation

Table 4.7 Incubation results for final full-scale inoculated pack studies (updated on June 13, 2011; after 80 days' incubation)

Process Level	F_0, min	Inoculum / Pouch	Replicate Runs	Number of		Observed Date (days)
				Sample Pouches	Positive Pouches [a]	
Control	N/A	5×10^3	N/A	30	30	1
Level-1	2.4	5×10^3	1	25	13	2 - 3
			2	25	14	2 - 3
		Un-inoculated	1	5	0	
			2	5	0	
Level-2	4.2	5×10^3	1	25	1	25
			2	25	1	2
		Un-inoculated	1	5	0	
			2	5	0	
Level-3	6.2	5×10^3	1	25	0	
			2	25	0	
		Un-inoculated	1	5	0	
			2	5	0	
Level-4	8.6	5×10^3	1	25	0	
			2	25	0	
		Un-inoculated	1	5	0	
			2	5	0	

a: When a complete bulging is detected, the pouch was considered positive.

5. Others

- Forty chicken dumpling pouches were processed with the MW processing schedule for F_0=6.2 min. After 10 days' incubation at 36.5°C, the pouches were shipped to Natick for evaluation.
- Forty chicken dumpling pouches were processed in hot water for F_0= 6 min and were shipped to Natick for evaluation after 10 days' incubation at 36.5°C.

General Summary

- The temperature profiles and F_0 values inside different food components at the cold spot identified by the chemical-marker-based computer-vision method inside chicken-dumpling pouches were measured with Ellab sensors. The chicken piece received the lowest thermal treatment and was chosen as the target component for temperature measurement in MW processing development.

- The temperature profiles and F_0 values at selected points inside chicken-dumpling pouches were measured with Ellab sensors. The cold spot identified by the chemical-marker-based computer-vision method was confirmed as the actual cold spot inside the real food pouch.

- Heat penetration (HP) test results show that, under the HP test conditions, the MW sterilization system delivered a thermal process to achieve F_0 higher than 6.0 min at the cold spot in chicken-dumpling pouches (each 8-oz PrintPack pouch filled with 95 g chicken breast pieces, 80 g sauce, 16 g dumplings, and 7 g vegetables) at the end of processing.

- Four processing schedules for inoculated pack studies were developed to achieve different target F_0 values varying from 2.4 to 8.6 min. An inoculation level of 5×10^3 CFU/pouch was selected for the inoculated pack studies.

- Preliminary inoculated pack studies were conducted in a small scale under three lower process levels (Level 1, 2, and 3).

- Full scale inoculated pack studies were finally conducted. Results showed that the developed MW sterilization processing delivered expected lethality to *C. sporogenes* PA 3679 spores.

- Forty chicken-dumpling pouches processed in MW for F_0=6.2 min and 40 pouches processed in hot water for F_0=6 min were shipped to Natick.

Future Work

- Prepare documentation for supporting USDA acceptance of the MW process for chicken-dumpling pouches.